

Effect of Various Concentrations of Sodium Hypochlorite on the Microhardness of Bulk-fill Restorative Composite Resin used as a Pre-endodontic Restoration

ANURADHA PATIL¹, PRIYANKA SANGLE², MV SUMANTHINI³, TANVI SATPUTE⁴, DIVYA NAIK⁵, ANTARA GHOSH⁶

ABSTRACT

Introduction: A four-walled access cavity plays a vital role in successful endodontic treatment. The materials used for pre-endodontic restoration are flowable composites, restorative composite resins, packable composite resins, silver amalgam, or glass ionomer cement. Researchers have claimed that exposure of composite resins to low pH liquids and root canal irrigants can have a deleterious effect on their physical and mechanical properties.

Aim: To evaluate the microhardness of a bulk-fill restorative composite resin material before and after being exposed to distilled water, 1% sodium hypochlorite, 3% sodium hypochlorite, and 5% sodium hypochlorite.

Materials and Methods: This was an in-vitro experimental study that was conducted at the Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Mumbai, Maharashtra, India, over a period of eight months from January 2021 till August 2021. Total 32 disc-shaped samples were made from a bulk-fill restorative composite resin, 3M™ Filtek Bulk fill posterior restorative composite resin (3M™, St. Paul, MN, USA). Each disc was prepared using polytetrafluoroethylene (Teflon) molds of 10 mm inner diameter and 4 mm depth. The bulk-fill composite resin discs were randomly divided into four main

groups, group 1 (distilled water), group 2 (1% NaOCl), group 3 (3% NaOCl), and group 4 (5% NaOCl), with eight samples in each group. Baseline Vickers hardness testing was performed for each group using a 100 gram load and a dwell time of 10 seconds before being immersed in the irrigation solution. Pre and post immersion microhardness measurements were done on the same surface of each sample (top surface). Data obtained were subjected to normality tests. Further statistical analysis was done using one-way Analysis of Variance (ANOVA) followed by the Games-Howell test for pair-wise comparisons.

Results: Irrespective of the different concentrations of sodium hypochlorite irrigant, all samples showed a reduction in microhardness of bulk-fill composite restorative resin. The post immersion microhardness mean values were highest in group 1 (63.06) and lowest in group 4 (58.42), and the difference was statistically significant ($p < 0.001$). No statistical difference was seen between group 3 and group 4. On intragroup comparison, all the groups show statistically highly significant difference between pre and post immersion microhardness values {Paired-t test ($p < 0.05$)}.

Conclusion: Microhardness of bulk-fill composite restorative resin was lowered by different concentrations of sodium hypochlorite irrigant.

Keywords: Dental resins, Materials testing, Permanent dental restorations, Root canal irrigants, Root canal therapy

INTRODUCTION

Microorganisms primarily mediate endodontic disease. The primary endodontic treatment goal should be directed toward minimizing the critical concentration of microbial irritants [1]. Thorough mechanical and chemical debridement of the root canal space results in successful endodontic outcomes. To preserve the functional integrity of teeth that have been destroyed by caries and require root canal treatment, a pre-endodontic build-up of the clinical crowns is often required [2]. Investment of time in placing pre-endodontic restoration can provide ease in rubber dam placement, aid in containing root canal irrigants, ensure a low probability of losing the provisional restoration, and improve the endodontic treatment prognosis [3].

Chemical debridement is essential for teeth with complex internal anatomies such as fins, cul de sac, ramifications, lateral and accessory canals, and other irregularities which might be overlooked by instrumentation [4]. By washing out debris, disintegrating tissue, and cleaning the root canal system, root canal irrigants can help with mechanical debridement [4-6]. Because of its antimicrobial effect and high efficiency in dissolving necrotic tissue, NaOCl is the most commonly recommended irrigating solution. [4]. Because of its proteolytic action, sodium hypochlorite can dissolve pulpal

remnants [7,8] and organic components of dentin [9]. It can also partially neutralise necrotic tissues as well as any antigenic or microbial component left in the root canal space [10]. Various concentrations of sodium hypochlorite ranging from 0.5-5.25% are widely used. Even though less concentrated solutions have shown antibacterial effectiveness, higher concentrations of NaOCl have a faster and more effective bactericidal effect. However, the higher the concentration of NaOCl, the greater the cytotoxic effect [11,12].

The materials used for pre-endodontic restoration are flowable composites, restorative composite resins, packable composite resins, silver amalgam, or glass ionomer cement [13,14]. Bulk-fill Resin Composite (BRCs) have been marketed with thickness of increments ranging from 4 to 10 mm [15]. The use of larger increments of BRCs shortens the time required to place posterior restorations and thus reduces technique sensitivity [16]. Resistance to wear and fracture is provided by the high filler loading [17,18].

Despite advancements in the composition and properties of restorative materials, pre-endodontic restorations are subjected to numerous root canal irrigants that may result in changes in their physical and mechanical properties like bond strength,

hardness, and fracture toughness [19-22]. Various studies have been performed to check for the effect of saliva, beverages, tea, coffee, and mouthwashes on the surface hardness of BRC's [23-31]. Researchers have claimed that exposure of composite resins to low pH liquids and root canal irrigants can have a deleterious effect on their properties [4]. In addition, several studies have evaluated a high propensity of composite materials for alteration when exposed to chemical substances such as hydrogen peroxide, organic acids, or ethanol [32-34]. To the best of our knowledge, no research has been done on how different sodium hypochlorite concentrations affect BRCs.

After completion of the endodontic treatment, the pre-endodontic restorations are often incorporated in the final post-endodontic restoration of the tooth [35]. Because the strength of this restoration is critical to the long-term success of the permanent restoration [26], the current study sought to assess the effect of various concentrations of sodium hypochlorite irrigant on the microhardness of BRC. The null hypothesis was that there is no difference in the effect of different concentrations of sodium hypochlorite irrigant on the microhardness of BRC.

MATERIALS AND METHODS

This experimental in-vitro study was carried out at the Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Maharashtra, India after obtaining the Institutional Ethics Committee approval (ethical clearance number: MGM/DCH/IRRC/6/2021). The study was conducted over a period of eight months (from January 2021 till August 2021).

Inclusion criteria: Samples prepared using 3M™ Filtek Bulk fill posterior restorative composite resin [3M™ Filtek, St. Paul, MN, USA] were included in the study.

Exclusion criteria: Samples that developed defects, errors during manipulation, or were damaged during finishing and polishing were excluded from the study.

Sample size calculation: Cochran's formula was used for calculating the sample size with the value of standard deviation as 1.41, reliability coefficient as 1.96, and the power of study considered was 80 [36].

Preparation of Composite Resin Samples

Samples were made using Teflon molds of 10 mm diameter and 4 mm height. Molds were placed on mylar strips placed on a glass slab and were filled with 3M™ Filtek Bulk fill posterior restorative composite resin [3M™ Filtek, St. Paul, MN, USA] according to the manufacturer's instructions. A mylar strip was placed on the upper surface of the mold, and the material was flattened with a microscope glass slide to achieve a standardised surface finishing and remove any excess material. After removing excess material, the glass microscope slide was removed, leaving the mylar strip. The Light Emitting Diode (LED) curing light (Ivoclar Vivadent AG, FL-9494 Schaan, Liechtenstein, Austria) was used to cure the samples for 40 seconds at an intensity of 1200 mW/cm², with the tip kept at a distance of 1 mm. An external handheld radiometer checked the intensity of the light curing unit before curing each sample.

After curing, the cured samples were separated from the molds. Samples were polished with Shofu Composite polishing kit (San Marcos, California, USA) using a low-speed handpiece with coolant according to the manufacturer's instructions. To achieve complete polymerisation, the samples were kept in an incubator at 37°C, 100% humidity in a lightproof container for 24 hours.

A total of 32 samples of BRC were prepared. The samples were randomly divided into four groups of eight samples each (n=08) as follows: group 1 [distilled water], group 2 [1% NaOCl], group 3 [3% NaOCl], group 4 [5% NaOCl].

Pre immersion Microhardness Testing

The baseline microhardness values of the samples were recorded using Vicker's microhardness tester (Microhardness tester, Reichert Austria Make, Sr.No.363798, Reference Standard: ISO 6507) for specimen indentation with a load of 100 g and a dwell time of 10 s. For each microhardness test, three indentations were made randomly on the top surfaces of each sample which were not closer than 1 mm to the adjacent ones. The average of the three readings was taken, and microhardness values were calculated. All hardness values were expressed in Vickers Hardness Number (VHN), where $1 \text{ VHN} = 1.854 \text{ P/d}^2$, with P being the indentation load and d being the diagonal length [37].

Post immersion Microhardness Testing

The immersion procedure was carried out by immersing the samples in test solutions and replenishing the solution every five minutes for 40 minutes. The most effective irrigation regimen to disinfect dentin was seen at 40 minutes, hence the samples were immersed in the test solutions for 40 minutes [38]. The samples were then rinsed with distilled water and dried using blotting/tissue paper. The post immersion microhardness was checked in the same manner as the baseline VHN determination. Pre and post immersion microhardness measurements were done on the same surface of each sample (top surface). Data were tabulated and statistically analysed.

STATISTICAL ANALYSIS

The data obtained were subjected to statistical analysis using MS Office Excel Sheet (v 2019, Microsoft Redmond Campus, Redmond, Washington, United States). First, the normality of data was tested using the Shapiro-Wilk test and was normally distributed ($p > 0.05$); hence parametric test (one-way ANOVA) was used for comparison followed by Games-Howell post hoc test Statistical Package for the Social Sciences software (SPSS v 26.0, IBM). An intra group comparison was made using paired t-test. The level of significance was set at 5% and keeping α error at 5% and β error at 20%, thus giving power to the study as 80%.

RESULTS

The control group, group 1 (Distilled water), had the highest mean microhardness values, and group 4 had the lowest mean microhardness values (NaOCl 5%) post immersion. All four groups' microhardness decreased upon immersion when compared to pre immersion values. Descriptive statistics of surface microhardness measurements for each experimental group are shown in [Table/Fig-1].

Group	n	Mean	Std. deviation	Std. error	95% Confidence interval for mean		Minimum	Maximum	F-value	p-value	
					Lower bound	Upper bound					
Pre immersion values	1	8	65.2675	3.5365	1.2503	62.3108	68.2241	58.9300	69.5900	0.528	0.667
	2	8	65.6562	1.2993	0.4593	64.5699	66.7425	63.7000	67.2300		
	3	8	65.3112	2.8762	1.0169	62.9066	67.7158	59.6800	69.2000		
	4	8	66.6300	1.3646	0.4824	65.4890	67.7709	64.3800	68.5300		

Post immersion values	1	8	63.0625	2.7851	0.9846	60.7340	65.3909	57.6000	66.5400	8.148	<0.001**
	2	8	59.9625	1.5318	0.5415	58.6818	61.2431	56.8000	61.5400		
	3	8	58.4375	2.4633	0.8709	56.3780	60.4969	54.1700	62.4100		
	4	8	58.4287	1.5911	0.5625	57.0985	59.7589	55.6500	60.1500		
Difference	1	8	2.2050	1.1251	0.3977	1.2643	3.1456	0.7400	3.8000	50.818	<0.001**
	2	8	5.6937	0.7897	0.2792	5.0335	6.3539	4.8200	7.2900		
	3	8	6.8737	0.7907	0.2795	6.2126	7.5348	5.5100	7.8000		
	4	8	8.2012	1.2844	0.4541	7.1273	9.2751	5.8100	9.7100		

[Table/Fig-1]: Descriptive statistics of VHN and intergroup comparison in all four groups. $p < 0.05$ was considered to be statistically significant; ANOVA; **: highly significant

The mean values of post immersion microhardness were higher in group 1 (63.06) followed by group 2 (59.96), group 3 (58.43) and least in group 4 (58.42). The ANOVA test showed a statistically highly significant difference in post immersion microhardness between the groups with $p < 0.01$ [Table/Fig-1].

Due to the non-homogeneity of variances ($p < 0.05$) [Table/Fig-2], Games-Howell post hoc test was applied. There was a significant difference in post immersion when group 1 was compared with group 3 ($p = 0.016$) and group 4 ($p = 0.008$) respectively. Also, statistical significance was seen when mean difference of group 2 was compared with group 3 ($p = 0.043$) and 4 ($p = 0.003$). However, no statistically significant differences were seen between group 3 and group 4 ($p = 0.114$) [Table/Fig-3].

Parameters		Statistics	df1	df2	Sig.
Pre immersion	Welch	0.937	3	14.748	0.447
	Brown-Forsythe	0.528	3	17.924	0.669
Post immersion	Welch	5.794	3	15.172	0.008
	Brown-Forsythe	8.148	3	22.484	0.001
Difference	Welch	38.166	3	15.288	<0.001
	Brown-Forsythe	50.818	3	23.785	<0.001

[Table/Fig-2]: Robust tests of equality of means.

Dependent variable	(I) group	(J) group	Mean difference (I-J)	Std. error	p-value	95% Confidence Interval	
						Lower bound	Upper bound
Pre immersion	1	2	-0.3887	1.3320	0.991#	-4.5610	3.7835
		3	-0.0437	1.6116	1.000#	-4.7529	4.6654
		4	-1.3625	1.3402	0.744#	-5.5427	2.8177
	2	3	0.3450	1.1158	0.989#	-3.0852	3.7752
		4	-0.9737	0.6662	0.485#	-2.9107	0.9632
	3	4	-1.3187	1.1255	0.657#	-4.7623	2.1248
Post immersion	1	2	3.1000	1.1238	0.076#	-0.2882	6.4882
		3	4.6250*	1.3145	0.016*	0.7967	8.4532
		4	4.6337*	1.1340	0.008**	1.2272	8.0402
	2	3	1.5250	1.0256	0.475#	-1.5312	4.5812
		4	1.5337	0.7808	0.247#	-0.7363	3.8038
	3	4	0.0087	1.0368	1.000#	-3.0704	3.0879
Difference	1	2	-3.4887*	0.4859	<0.001**	-4.9222	-2.0552
		3	-4.6687*	0.4862	<0.001**	-6.1027	-3.2347
		4	-5.9962*	0.6037	<0.001**	-7.7548	-4.2376
	2	3	-1.1800*	0.3951	0.043*	-2.3283	-0.0316
		4	-2.5075*	0.5330	0.003**	-4.0977	-0.9172
	3	4	-1.3275	0.5332	0.114#	-2.9181	0.2631

[Table/Fig-3]: Games-Howell post hoc test for Inter group comparison of mean Vicker's microhardness.

* = statistically significant difference ($p < 0.05$) ** = statistically highly significant difference ($p < 0.01$)

= non-significant difference ($p > 0.05$)

An intra group comparison was made using paired t-test which showed statistically highly significant ($p < 0.01$) difference in the pre and post immersion microhardness values of all the four groups [Table/Fig-4].

DISCUSSION

The quality of the instrumentation, irrigation, disinfection, obturation, and lastly the coronal seal of the root canal system completely determines the long-term prognosis of endodontic treatment. The significance of the coronal seal has been increasingly recognized in the dental literature. In more recent times, it has been proposed that coronal leakage is much more likely to be the key determinant of clinical success or failure than apical leakage [39]. Amalgam, glass ionomers, and composites are commonly used pre-endodontic build-up materials in today's practice [40]. The use of composites as build-up materials should be favored due to the known drawbacks of amalgam as a build-up material (corrosion and staining of the remaining tooth structure) and the low mechanical properties of glass ionomers in comparison to composites [2,3,5,6].

The effect of irrigating solutions on the pre-endodontic restoration during irrigation may change the physical and chemical properties of the restorative material, including hardness [41,42]. Hence, the present study was carried out to evaluate the effect of various concentrations of NaOCl endodontic irrigating solutions, i.e., distilled water, 1% NaOCl, 3% NaOCl, and 5% NaOCl on the microhardness of a BRC. Contact with sodium hypochlorite on build-up materials has previously been shown to reduce their microhardness.

The softening can be found in deeper layers of build-up materials and not only limited to the surface [43]. Microhardness of bulk-fill composites is also altered after exposure to various agents like bleaching agents [44]. Microhardness of bulk-fill composites has also

Variables		Mean	Std. Deviation	Std. Error Mean	Mean diff	SD of diff	95% lower bound confidence interval of the difference	95% upper bound confidence interval of the difference	T value	p-value of paired t-test
Group 1	Pre	65.2675	3.5365	1.2503	2.2050	1.1251	1.2643	3.1456	5.543	0.001**
	Post	63.0625	2.7851	0.9846						
Group 2	Pre	65.6562	1.2993	0.4593	5.6937	0.7897	5.0335	6.3539	20.393	<0.001**
	Post	59.9625	1.5318	0.5415						
Group 3	Pre	65.3112	2.8762	1.0169	6.8737	0.7907	6.2126	7.5348	24.588	<0.001**
	Post	58.4375	2.4633	0.8709						
Group 4	Pre	66.6300	1.3646	0.4824	8.2012	1.2844	7.1273	9.2751	18.059	<0.001**
	Post	58.4287	1.5911	0.5625						

[Table/Fig-4]: Comparison of mean values of Vicker's microhardness of pre and post immersion in all groups.

p<0.01 considered significant for intragroup comparison; paired t-test

been shown to be affected after immersion in different solutions like coffee, cola, red wine, and distilled water [45]. The BRC was chosen for this study since the material simplifies the clinical procedure and randomized controlled studies have proven them to be successful [46,47] however, information with regards to their performance in restoring root-filled teeth is inadequate.

Micro hardness testing is one of the most simple and non destructive methods for studying fine-scale changes in a material's hardness [48]. The Vickers hardness test is one method for measuring micro hardness. The Vickers hardness test was used in this study because it is an appropriate and practical method for evaluating changes on the surface and in deeper hard tissue structures [49]. Furthermore, this test is widely accepted due to its highly accurate readings and the fact that only one type of indentation is used in this method for all types of surface treatment [50].

In the present study, the samples were polished and finished with Shofu [San Marcos, California, USA] to remove any surface imperfections and achieve a mirror-like quality. When using the VHN testing equipment, the glossy surface promotes light reflection so that indentations may be seen clearly. To achieve maximum polymerization, the specimens were stored in an incubator for 24 hours immediately after fabrication before being immersed in the experimental solution.

For standardisation, all samples were immersed for 40 mins in the selected irrigating solutions and the solution was replenished every 5 min to mimic the clinical scenario.

An excellent restorative material should exhibit good mechanical properties to replace natural tooth tissue. During endodontic treatment, dental composites are exposed intermittently or continuously to chemical agents, which could cause chemical degradation and loss of many dental properties [22].

In the present study, it was observed that the microhardness of BRC is inversely proportional to the concentration of the irrigants, thus rejecting the null hypothesis. An alteration in the composition of the restorative material may be responsible for the decrease in microhardness. Hypochlorite and hypochlorous acid, the reactive chlorine derivatives of sodium hypochlorite, exhibit significant oxidizing potential [51]. It is seen that substrates with oxidative capabilities may break polymer chains, leading to the decomposition of resin-based materials thus affecting their properties [32]. Oxidative substrates may also have an impact on the hardness of composites by contributing to debonding of the filler matrix [51]. Therefore, before the final prosthetic restoration, it appears that alteration of the composite brought on by contact with NaOCl necessitates a total replacement of the build-up.

Limitation(s)

The in-vitro environment does not accurately represent the natural conditions of the oral cavity. As a result, future research should test more experimental designs that can depict the clinical behaviour of the restorative material in-vitro.

CONCLUSION(S)

It can be concluded that contact of sodium hypochlorite with bulk-fill restorative composite resin causes a reduction in microhardness. Alteration in the properties of the BRC can compromise the coronal seal along with the ability to sustain the masticatory forces directed towards the endodontically treated tooth to achieve the desired outcome of the restorative treatment. Therefore it is deemed necessary to completely replace the pre-endodontic restoration prior to the definitive prosthetic restoration for its longevity. Future studies have to be done with different irrigating solutions with different core build-up materials and various material properties such as sorption, solubility, and nanoleakage should be checked. Furthermore, to overcome the limitations, further advances are to be made in materials to enhance the physical and mechanical properties of the pre-endodontic restoration material.

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PARTICULARS OF CONTRIBUTORS:

1. Professor, Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Mumbai, Maharashtra, India.
2. Former Fellowship Student, Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Mumbai, Maharashtra, India.
3. Professor and Head, Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Mumbai, Maharashtra, India.
4. Assistant Professor, Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Mumbai, Maharashtra, India.
5. Associate Professor, Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Mumbai, Maharashtra, India.
6. Assistant Professor, Department of Conservative Dentistry and Endodontics, M.G.M Dental College and Hospital, Kamothe, Navi Mumbai, Mumbai, Maharashtra, India.

NAME, ADDRESS, E-MAIL ID OF THE CORRESPONDING AUTHOR:

Anuradha Patil,
Junction of NH4 and, Sion - Panvel Expy, Sector 18, Navi Mumbai, Maharashtra
410209, Mumbai, Maharashtra, India.
E-mail: anuradhapatil32@gmail.com

PLAGIARISM CHECKING METHODS: [Jain H et al.]

- Plagiarism X-checker: May 05, 2022
- Manual Googling: Aug 05, 2022
- iThenticate Software: Aug 08, 2022 (22%)

ETYMOLOGY: Author Origin

AUTHOR DECLARATION:

- Financial or Other Competing Interests: None
- Was Ethics Committee Approval obtained for this study? Yes
- Was informed consent obtained from the subjects involved in the study? No
- For any images presented appropriate consent has been obtained from the subjects. NA

Date of Submission: **May 02, 2022**

Date of Peer Review: **May 28, 2022**

Date of Acceptance: **Aug 09, 2022**

Date of Publishing: **Oct 01, 2022**