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Original Article

Dentistry Section

Effect of Three Different Concentrations of Sodium Hypochlorite on the Solubility of Bulk Fill Restorative Composite Resin: An In-vitro Study

ANURADHA PATIL[1], SNEHAL KALSEKAR[2], MV SUMANTHINI[3], DIVYA NAIK[4], ADITYA KIRAN SHINDE[5], SHRESHTHA MUKHERJEE[6]

ABSTRACT

Introduction: The integrity of pre-endodontic restorations plays a vital role in ensuring the longevity of permanent restorations, particularly in cases involving extensive tooth structure loss. Sodium Hypochlorite (NaOCl) is commonly used as a root canal irrigant due to its antimicrobial properties, but its potential effects on the solubility of restorative materials, such as bulk fill composites, remain underexplored. Understanding how varying concentrations of NaOCl impact these materials is essential for optimising endodontic treatment outcomes and enhancing the durability of subsequent restorations.

Aim: To evaluate the effect of three different concentrations of NaOCl as a root canal irrigant on the solubility of bulk fill restorative composite resin.

Materials and Methods: This in-vitro study was carried out at the Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Kamothe, Navi Mumbai, Maharashtra, India, over a period of nine months from August 2023 till April 2024. Total 24 disc-shaped samples were prepared using polytetrafluoroethylene moulds of 10×4 mm of Tetric N Ceram bulk fill restorative composite resin. Samples were randomly divided into Group I (distilled water), Group II (1% NaOCl), Group III (3% NaOCl), and Group IV (5% NaOCl), with six samples in each group. Solubility tests were performed according to ISO 4049. Analysis of Variance (ANOVA) followed by Tukey's Post-hoc test was applied to compare solubility between and within groups.

Results: The mean solubility values were highest in Group IV (-1.16±0.26 μg/mm^3) and lowest in Group I (-0.36±0.13 μg/mm^3), with the difference being statistically highly significant ($p<0.001$). No statistically significant difference was observed between Group I (distilled water) and Group II (1% NaOCl) ($p>0.05$). There was also no statistically significant difference when Group II was compared with Group III (3% NaOCl) and Group I (distilled water), respectively ($p>0.05$).

Conclusion: Bulk fill composite resin exhibits increased solubility post immersion in higher concentration of NaOCl (5%). To minimise degradation and ensure restoration longevity, 1-3% NaOCl concentration is recommended. However, due to the compromised integrity of the resin, it is recommended to replace the pre-endodontic restoration before proceeding with permanent post-endodontic restoration to prevent potential coronal leakage and ensure the success of the final restoration.

Keywords: Permanent dental restorations, Polytetrafluoroethylene, Root canal irrigants, Root canal therapy

INTRODUCTION

Endodontic therapy aims to completely remove bacteria, microbial biofilms, and their by-products from the root canal system through a process of chemo-mechanical debridement. This approach is crucial for preventing further contamination of intracanal spaces, as microorganisms are recognised as the primary contributors to endodontic disease. However, before initiating endodontic treatment, it is essential to address certain structural issues of the tooth [1].

Teeth requiring endodontic treatment often have compromised structural integrity due to factors such as caries, trauma, or root resorption [2]. Therefore, pre-endodontic restorations become necessary to address these issues. These restorations facilitate optimal rubber dam isolation, create space for extended irrigation solution function, and enable effective interim temporisation between appointments. This prevents bacterial leakage and seepage of intracanal medicaments, reduces the risk of gingival ingrowth into the cavity, and helps prevent fractures in weakened tooth structures. Moreover, pre-endodontic built-up facilitates post-endodontic restoration procedures, making it a critical step in the overall treatment plan [3,4].

In terms of materials used for pre-endodontic restoration, there is a range of options, including flowable composites, packable composite resins restorative composite resins, silver amalgam, and glass ionomer cement [4]. Among these, bulk-fill composite resin is often preferred due to its improved handling properties, reduced polymerisation shrinkage, and enhanced durability. Studies have demonstrated that bulk-fill composites exhibit lower shrinkage stress and better marginal adaptation compared to conventional composites [5]. They also show superior durability and resistance to fracture under occlusal loading. Additionally, bulk-fill composites offer benefits such as simplified placement, reduced technique sensitivity, and improved clinical outcomes. With increments ranging from 4 to 10 mm in thickness, Bulk-fill Resin Composites (BRCs) provide an optimal choice for pre-endodontic restoration, ensuring both improved performance and clinical efficiency [6,7].

Following pre-endodontic restoration, the next crucial aspect of endodontic therapy involves the use of effective irrigants [8]. NaOCl is a pivotal irrigant in this context, known for its potent antimicrobial properties and its ability to dissolve organic tissue. Its significance lies in the fact that mechanical instrumentation alone often fails to remove pulpal tissue from complex canal anatomies, such as oval extensions, isthmuses, and irregularities [9]. Commonly used concentrations of NaOCl in dentistry range from 0.5 to 5.25%. Recommendations emphasise the importance of frequent irrigation exchanges and increased volume to maximise efficacy [10]. While lower concentrations are effective against bacteria, higher concentrations offer a faster and more potent bactericidal effect, though with an increased risk of cytotoxicity [11,12].

Solubility, another crucial parameter, refers to the dissolution of resins in oral fluids. Understanding the solubility of restorative materials is crucial for anticipating their behaviour in the oral environment. Solubility significantly influences the integrity, mechanical properties, surface characteristics, and aesthetic appearance of restorations [13].

During endodontic therapy, pre-endodontic restorations are exposed to various root canal irrigants for different intervals, which affect their physical and mechanical properties. This exposure leads to changes in hardness, bond strength, and fracture toughness due to the leaching of ingredients [14]. Previous studies have investigated the impact of various substances on bulk-fill composite resins, including saliva, food-simulating liquids, cigarette smoke, and mouthwashes [13-18]. These studies have shown that exposing composite resins to low-pH liquids and root canal irrigants can adversely affect their properties [19,20].

Regarding NaOCl, it is essential to consider its effects on pre-endodontic restorations. NaOCl is a common root canal irrigant, and its impact on composite resins should be understood. Although some studies have investigated the impact of NaOCl on bulk-fill composite resins, there is a lack of comprehensive research evaluating the effects of different concentrations of NaOCl as a root canal irrigant on the properties of bulk-fill composite resins, particularly in terms of solubility [21,22]. The present study is aimed to evaluate the effect of three different concentrations of NaOCl as a root canal irrigant on the solubility of bulk-fill restorative composite resin. The null hypothesis is that there is no difference in the effect of different concentrations of NaOCl as a root canal irrigant on the solubility of BRC.

MATERIALS AND METHODS

This in-vitro study was carried out at the Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Kamothe, Navi Mumbai, Maharashtra, India, after obtaining approval from the Institutional Ethics Committee (ethical clearance number: MGM/DCH/IEC/193/2023). The study was conducted over a period of nine months (from August 2023 to April 2024).

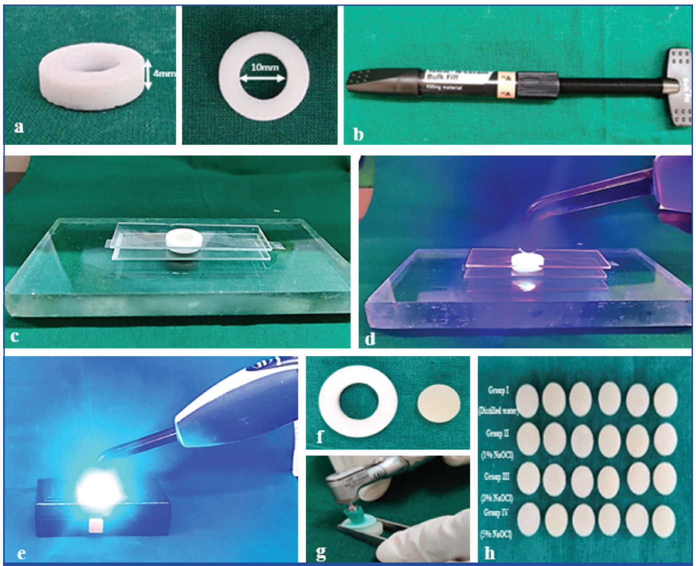
Inclusion criteria: Samples prepared using Ivoclar Vivadent Tetric N-Ceram Bulk Fill restorative composite resin (Ivoclar Vivadent AG, FL-9494 Schaan, Liechtenstein, Austria) were included in the study.

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

Sample size calculation: Sample size was determined using G*Power software 3.1 and Bernard Rosner formula [23], with an effect size of 2.75 [16] and power of the study set at 80% [16].

Study Procedure

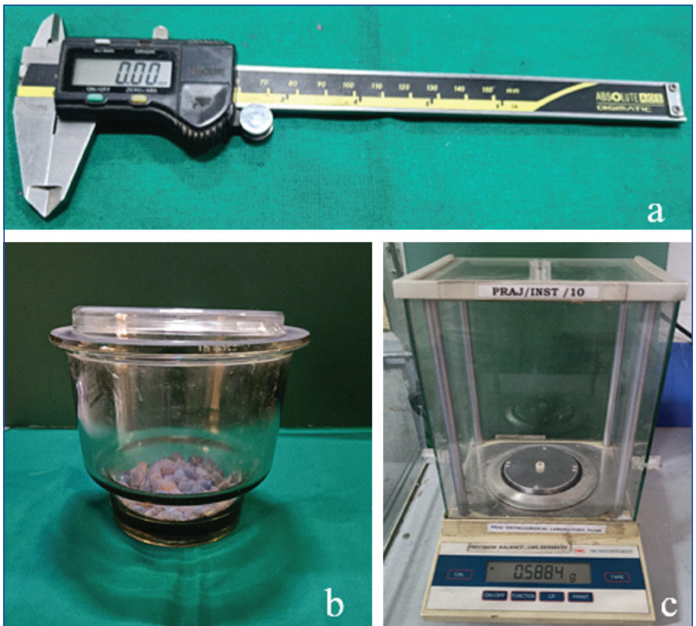
Preparation of composite resin samples: Samples were made using Teflon moulds of 10 mm diameter and 4 mm height. Moulds were placed on a glass slab, and a single increment of Tetric N-Ceram Bulk-Fill Restorative Composite Resin (Ivoclar Vivadent AG, FL-9494 Schaan, Liechtenstein, Austria) was packed into the moulds according to the manufacturer's instructions. A mylar strip was placed on the upper surface of the mould, and the material was flattened with a glass microscope slide to achieve a standardised surface finishing and remove any excess material. The Ivoclar Bluephase NMC Light-emitting Diodes (LED) curing light, emitting 1200 mW/cm², was utilised for 40 seconds to cure the samples, maintaining a 1 mm distance. Prior to each curing cycle, the Bluephase radiometer verified the light intensity [Table/Fig-1].



[Table/Fig-1]: Preparation of composite resin samples: a) Teflon moulds of 10 mm diameter and 4 mm height; b) Tetra-N-Ceram bulk-fill composite resin; c) Preparation of composite discs between glass slides; d) Composite resin cured with curing lamp; e) Measuring light intensity with digital radiometer; f) Composite resin discs removed from the mould; g) Samples finished and polished using Shofu's finishing and polishing kit; h) Samples randomly divided into four groups of six samples each group.

Post-curing, samples were removed from molds and polished using Shofu's composite polishing kit with a low-speed handpiece and coolant, adhering to manufacturer's guidelines. Total 24 BRC samples were prepared and randomly divided into four groups (n=6): Group I (distilled water), Group II (1% NaOCl), Group III (3% NaOCl), and Group IV (5% NaOCl). Samples were stored in light-proof containers and incubated at 37°C±1°C, at 100% humidity for 24 hours to ensure complete polymerisation [14], and were stored in light-proof containers.

Solubility measurements: Solubility measurements were conducted in line with International Organisation for Standardisation (ISO) 4049 standards. Samples were inserted into desiccator and maintained at 37±1ºC for 22 hours, followed by an additional two hours at 23±1ºC, and then weighed on an analytical scale accurate up to 0.0001 g (Precision Balance, LWL Germany, Model: LB-210S; accuracy: 0.0001 g) [Table/Fig-2]. This procedure was repeated until a constant mass was obtained, and values were recorded as M0. A



[Table/Fig-2]: Solubility measurement: a) Digital Calliper (Digital Micrometer, Mitutoyo, Japan, Sr. No.293-821; accuracy=0.001 mm) was employed for measuring the diameter and thickness; b) Vacuum desiccator. Samples were inserted into desiccator and maintained at 37±1ºC for 22 hours and at 23±1ºC for additional two hours; c) Samples after desiccation were weighed on analytical scale accurate up to 0.0001 g (Precision Balance, LWL Germany, Model: LB-210S; accuracy: 0.0001 g).

digital caliper (Digital Micrometer, Mitutoyo, Japan, Sr No. 293-821; accuracy=0.001 mm) was employed for measuring the diameter and thickness. To determine the dimensions, two perpendicular diameter measurements were taken, and the average value was computed. Thickness was assessed at the center and four equidistant circumferential points, yielding a mean thickness.

Specimen volume (V) was calculated in cubic millimeters using the formula: $V=\pi \times (diameter/2)^2 \times$ mean thickness, where radius (r) was derived from the diameter and height (h) represented the mean thickness [16-18].

The immersion procedure was carried out by immersing the samples in test solutions and replenishing the solution every five minutes for 40 minutes [24]. From the total of 24 samples prepared, six samples were immersed in each irrigating solution, i.e., distilled water, 1% NaOCl, 3% NaOCl, and 5% NaOCl. Distilled water served as the control group. Samples were extracted and desiccated again as described for M0 until a constant mass was obtained, and the weights were recorded again (M1). Solubility (SL) was recorded in µg/mm³ as the change in weight before and after immersion using the formula: SL=(M0-M1)/V, where V is the volume of the sample in mm³ [16-18].

STATISTICAL ANALYSIS

Statistical analysis was done with Statistical Package for Social Sciences (IBM SPSS Statistic for window, version 21.0; Armonk, NY: IBM Corp.) with a 95% Confidence Interval (CI) and 80% power for the study. Normal distribution of the data was checked using Shapiro-Wilk test. Descriptive statistics were performed in terms of mean and standard deviation. ANOVA test indicated a statistically significant and followed by Tukey's Post-hoc test was applied to compare solubility between and within groups. Statistical significance was calculated at p<0.05.

RESULTS

The control group, Group I (distilled water), had the lowest solubility, while Group IV (5% NaOCl) had the highest solubility post immersion. All four groups weights increased upon immersion when compared to pre-immersion values. The ANOVA test showed a highly significant difference in solubility between the groups (p<0.001) [Table/Fig-3].

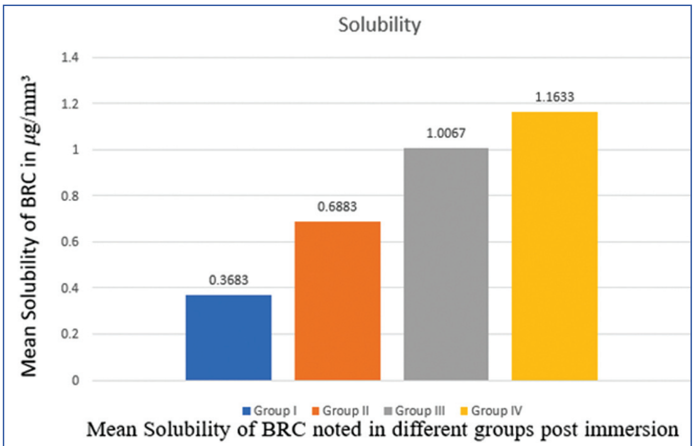
Groups	N	Mean±SD	Minimum	Maximum	F value	p-value
Group I (distilled water)	6	-0.3683±0.13318	0.31	0.64	14.951	<0.001*
Group II (1% NaOCl)	6	-0.6883±0.12828	0.63	0.95		
Group III (3% NaOCl)	6	-1.0067±0.31194	0.64	1.59		
Group IV (5% NaOCl)	6	-1.1633±0.26128	0.95	1.59		

[Table/Fig-3]: Descriptive statistics and intergroup comparison of solubility in all four groups.
*The mean difference is significant at the 0.05 level

Due to the non homogeneity of variances (p<0.05), Tukey's Post-hoc test was applied. In pairwise comparisons, it was observed that Group I (distilled water) showed a statistically significant difference in solubility compared to Group III (3% NaOCl) and Group IV (5% NaOCl) (p<0.001). No statistically significant difference was observed between Group I (distilled water) and Group II (1% NaOCl) (p>0.05). Group II (1% NaOCl) showed a statistically significant difference in solubility when compared with Group IV (5% NaOCl) (p<0.05). There was no statistically significant difference observed when Group II was compared with Group III (3% NaOCl) and Group I (Distilled Water) (p>0.05) [Table/Fig-4]. The mean solubility was highest in Group IV (1.1633), followed by Group III (1.0067), Group II (0.6883), and Group I (0.3683) [Table/Fig-5].

(I) Groups	(J) Groups	Mean difference (I-J)	p-value	95% Confidence interval Lower bound	95% Confidence interval Upper bound
Group I (distilled water)	Group II	0.32000	0.094	-0.6811	0.0411
	Group III	0.63833*	<0.001*	-0.9995	-0.2772
	Group IV	0.79500*	<0.001*	-1.1561	-0.4339
Group II (1% NaOCl)	Group I	-0.32000	0.094	-0.0411	.6811
	Group III	0.31833	0.096	-0.6795	.0428
	Group IV	0.47500*	0.007*	-0.8361	-0.1139
Group III (3% NaOCl)	Group I	-0.63833*	<0.001*	0.2772	0.9995
	Group II	-0.31833	0.096	-0.0428	0.6795
	Group IV	0.15667	0.625	-0.5178	0.2045
Group IV (5% NaOCl)	Group I	-0.79500*	<0.001*	0.4339	1.1561
	Group II	-0.47500*	0.007*	0.1139	0.8361
	Group III	-0.15667	0.625	-0.2045	0.5178

[Table/Fig-4]: Intergroup pair-wise comparison of solubility between groups.



[Table/Fig-5]: Graph showing mean solubility of BRC in Group I (distilled water), Group II (1% NaOCl), Group III (3% NaOCl) and Group IV (5% NaOCl) with X-axis representing solubility of BRC in µg/mm³ and Y-axis representing the solubility of BRC noted in different groups post immersion.

DISCUSSION

The quality of chemical and mechanical disinfection, obturation, and, ultimately, the coronal seal of the root canal system significantly influences the long-term prognosis of endodontic treatment. The significance of the coronal seal is becoming more widely recognised in the dental literature. In more recent discussions, it has been argued that the key factor influencing clinical outcomes—whether successful or not is coronal leakage rather than apical leakage [25,26].

Endodontic treatment is often needed for teeth with structural compromises, which can complicate the procedure. Thus, practitioner should prioritise pre-endodontic restoration as a crucial step for these cases. It is extremely important to reinforce structures such as marginal ridges and cusps, as they are mainly responsible for the resistance of teeth. Their reconstruction will aid in maintaining the integrity of the remaining teeth [2,3].

Traditionally, non adhesive materials and techniques, such as amalgam core buildup, copper/orthodontic bands with temporary cements, and temporary crowns, were utilised for pre-endodontic built-up, each with its own advantages and disadvantages [2]. However, these methods are now considered obsolete. Contemporary approaches involve the use of adhesive restorative materials like flowable composite, bulk-fill composite, and Resin-Modified Glass Ionomer Cement (RMGIC), employing various techniques such as cervical margin relocation, doughnut technique, canal projection, and open sandwich technique [27].

Additionally, surgical or orthodontic techniques, like surgical crown lengthening, orthodontic extrusion, and surgical extrusion, are employed. Restorations performed with adhesive materials offer the advantage of bonding to the tooth structure, potentially strengthening it and providing an alternative to indirect restoration methods [2,27].

The BRC simplifies clinical procedures and has demonstrated success in randomised controlled studies. However, their effectiveness as pre-endodontic restorations has not been thoroughly explored. Variations in water sorption and solubility among dental composites stem from differences in filler types and organic matrix compositions. Certain monomers' hydrophilic nature increases water absorption, with Triethylene Glycol Dimethacrylate (TEGDMA) (69.51 μg/mm³) exhibiting the highest sorption, followed by Bisphenol A-glycidyl Methacrylate (Bis-GMA) (33.49 μg/mm³), Urethane Dimethacrylate (UDMA) (29.46 μg/mm³), and Ethoxylated bisphenol A Dimethacrylate (Bis-EMA) (20.10 μg/mm³). Tetric N Ceram's higher filler content and inclusion of Bis-GMA, UDMA, and Bis-EMA monomers contribute to enhanced water absorption properties [17].

In present study, the hypothesis was tested to determine if different concentrations of NaOCl affect the solubility of BRC. The null hypothesis was rejected, indicating a significant effect of NaOCl concentration on BRC solubility, with higher concentrations leading to greater solubility.

Solubility of restorative materials, such as bulk-fill composite resins, can have a cascading effect on various other properties, ultimately impacting the longevity and success of endodontic restorations [28]. High solubility can lead to decreased mechanical properties, reduced bonding to tooth structure, increased water sorption, release of toxic components, an increased risk of secondary caries and bacterial penetration, and compromised optical properties. These factors can potentially cause a decrease in flexural strength and hardness, leading to debonding and microleakage, swelling, softening, and degradation of the material, irritation of pulp or periradicular tissues, and negatively affecting aesthetic appearance [29,30]. These interconnected effects highlight the importance of evaluating solubility in the context of endodontic restorations, as it can have far-reaching consequences for the durability and success of the treatment.

The NaOCl is known for its strong oxidising properties due to the formation of reactive chlorine compounds, hypochlorite and hypochlorous acid. These compounds can significantly affect resin-based dental products by disrupting polymer chains, leading to composite degradation and changes in material characteristics [19,25]. Research by Saleh AA and Ettman WM indicates that NaOCl can also cause debonding of filler particles from the resin matrix, affecting the composite's hardness [30].

Higher concentrations of NaOCl are associated with increased cytotoxicity and adverse effects on dental tissues, including dentin erosion, collagen degradation, and decalcification, all of which pose significant challenges to the structural integrity of dental tissues [31,32]. In the realm of antimicrobial efficacy, the effectiveness of NaOCl in eradicating intracanal microbiota has yielded inconsistent findings [33]. Some studies suggest that higher concentrations do not significantly improve bacterial eradication compared to lower concentrations [31-33]. For effective disinfection, lower concentrations of NaOCl may be sufficient without compromising clinical outcomes [33,34].

To standardise the process, immersion in various NaOCl solutions lasted 40 minutes, with replenishment every five minutes to simulate clinical conditions. Retamozo B et al., suggested a minimum irrigation time of 40 minutes for effective removal of *E. faecalis* from infected dentin; hence, the samples were immersed for the same duration [24].

Samples were polished with the Shofu composite finishing and polishing kit (San Marcos, California, USA) to eliminate surface imperfections and ensure a mirror-like finish. This step minimised variation in solubility measurements. Specimens were then incubated at 37±1ºC for 24 hours post-fabrication to ensure optimal polymerisation and stability for testing.

Dessication was carried out before and after immersion to eliminate surface moisture, stabilise the material, and ensure accurate solubility measurements. This process standardises testing conditions and minimises sample variability.

The present study observed an increase in weight following immersion in various concentrations of NaOCl, indicating the solubility of the BRC composite. The weight gain is attributed to the composite's absorption characteristics, where solvent permeates the polymer matrix and filler particles, causing swelling and thus an increase in weight. This swelling can be due to the solvent interacting with the resin matrix and filler particles, leading to a temporary expansion of the composite material [35,36].

Additionally, the Hypochlorite ion (OCl^-) may react with the resin's organic components, potentially forming new compounds that further contribute to the weight gain. This interaction and absorption effect can lead to negative solubility values, as the net increase in weight overshadows the potential dissolution of the resin. Such negative values reflect the composite's tendency to absorb and retain water or other solvents rather than fully dissolving [29].

Solubility quantifies the release of residual unconverted monomer into the solution, which is critical for assessing material stability. Negative solubility values in this context may stem from water trapped within the polymeric structure during storage or incomplete dehydration. This suggests minimal solubility rather than complete insolubility [37]. Lopes LG et al., noted that negative solubility values might arise from heightened water sorption in resin composites, which increases mass and can obscure the actual solubility measurement [28].

According to ISO 4049, solubility should not exceed 7.5 μg/mm³ [36,38]. The solubility values observed in this study fall within this acceptable range, indicating that the bulk-fill restorative composite resin compliance with ISO 4049 standards and is suitability for dental restorations. Nonetheless, solubility is only one aspect of material performance. Even within acceptable limits, excessive solubility could affect other properties, such as structural stability and marginal integrity [39]. Thus, evaluating solubility in conjunction with other performance factors, including hardness and durability, is essential for a comprehensive assessment of material suitability.

Limitation(s)

Restorations are subjected to varying temperatures and mechanical stresses, such as chewing and tooth brushing, which can affect composite solubility values. The present study's limitation is its inability to fully replicate the oral environment. Future research should explore how solubility variations influence the longevity and performance of dental restorations, and explore strategies to mitigate potential negative effects. Additionally, studies should focus on developing new materials and formulations that balance between superior mechanical properties with chemical stability. Long-term clinical evaluations are crucial to assessing the performance of restorative materials in real endodontic conditions and their durability against chemical degradation.

CONCLUSION(S)

The study demonstrates a concentration-dependent increase in solubility of bulk-fill restorative composite when exposed to NaOCl, with 5% NaOCl showing higher solubility than 1% and 3%. It is recommended to use lower concentrations of NaOCl (1-3%) in clinical practice, alongside optimised irrigation protocols. These protocols should include increasing the volume of irrigant used, extending the irrigation time, utilising passive ultrasonic activation or other agitation techniques, and ensuring adequate flushing and removal of irrigant. Although the solubility observed in this study complies with ISO 4049 standards, replacing pre-endodontic restorative material before final prosthetic restoration is still advisable, as even minimal solubility may affect the overall performance and longevity of the restorative material. By adopting these strategies, clinicians can enhance long-term success and improve restoration resilience and lifespan.

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PARTICULARS OF CONTRIBUTORS:
1. Professor, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Navi Mumbai, Maharashtra, India.
2. Fellowship Student, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Navi Mumbai, Maharashtra, India.
3. Professor and Head, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Navi Mumbai, Maharashtra, India.
4. Reader, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Navi Mumbai, Maharashtra, India.
5. Reader, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Navi Mumbai, Maharashtra, India.
6. Lecturer, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Navi Mumbai, Maharashtra, India.

NAME, ADDRESS, E-MAIL ID OF THE CORRESPONDING AUTHOR:
Dr. Anuradha Patil,
Professor, Department of Conservative Dentistry and Endodontics, MGM Dental College and Hospital, Junction of NH4 and Sion-Panvel HWY, Sector 18, Navi Mumbai-410209, Maharashtra, India.
E-mail: anuradhapatil32@gmail.com

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