ORIGINAL ARTICLE

Cocos nucifera L. (coir) fiber application as a filler and its effect on the volumetric shrinkage of flowable composite resin: an in vitro study

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ABSTRACT

Introduction: The use of composite resin as a restorative material has the disadvantage of experiencing volumetric shrinkage during polymerization, which can lead to restoration failure. Fiber-reinforced composites can reduce volumetric shrinkage in restorative materials. Coir fiber has the potential to replace synthetic fillers because its hollow fiber structure resembles foam, supporting a compact yet lightweight structure. This characteristic is expected to reduce the volumetric shrinkage of composite resin. This study aims to analyze the effect of coir fiber application as a filler on flowable composite resin on shrinkage, comparing it with flowable composite resin containing synthetic filler. **Methods:** The cellulose fiber used as a filler was synthesized from coir, while the matrix consisted of BisGMA and TEGDMA resins in a ratio 5:1. The composite was prepared with a coir fiber-to-matrix resin composition of 70:30, and polymerization was carried out by irradiation for 40 seconds. Volumetric shrinkage was tested 90 minutes after irradiation. The dimensions of the composite sample were measured from images captured using a digital microscope and analyzed with ImageJ software. The volume shrinkage was then calculated based on these dimensions. Shrinkage was further confirmed through SEM analysis of marginal adaptation tests. **Results:** The application of coir fiber as a filler showed the volumetric shrinkage of 0.0064% compared to the synthetic filler of 0.0401%. Statistical analyses using the Mann-Whitney test results indicated that the type of filler in flowable composite resin had a significant effect, with a p-value of 0.047 ($p<0.05$). **Conclusion:** Application of coconut fiber as filler in flowable composite resin significantly reduces volumetric shrinkage, making it three times smaller than that of flowable composite resin with a synthetic filler. This study supports environmentally friendly dental composites, enhancing the lifespan of restorations by assessing shrinkage cost-effectively.

KEYWORDS
Composite, cocos nucifera L., fiber filler, flowable, volumetric shrinkage

INTRODUCTION

Composite resin is the most widely used dental restorative material and the gold standard for restoring both anterior and posterior teeth.[1] Its advantages include biocompatibility, the absence of mercury, resistance to dissolution in saliva, the ability to bond with the hard tissue structure of the teeth, aesthetic appeal, and cost- and time-efficiency due to its ease of of direct application into cavities.[1,2] A significant disadvantage of composite resin is its tendency to shrink during polymerization.

Polymerization is the process of forming polymers by combining several monomers through radical groups, which are generated by chemical activation (in self-cured composite) or by light exposure (in light-cured composite).[2] Composite resin generally shows a volumetric shrinkage ranging from 1% to 6%. [3,4] Even a minimal volumetric shrinkage of 2% can generate shrinkage stress, leading to the failure of composite adhesion to dentin. Similarly, a linear shrinkage of 0.1 to 2% can result in maximum shrinkage stress ranging from 5 to 100 MPa.[5]

A previous study conducted by Abbasi et al. found no significant difference in polymerization shrinkage between in bulk-fill composite resin and conventional composite resin.[6] In contrast, Lins et al. reported differences in linear shrinkage values among the packable composite resins tested.[7] Similarly, Silva et al. observed variations in volumetric shrinkage values across conventional, bulk-fill, and low-shrinkage composite resins.[8] These studies collectively indicate that composite resins from different brands exhibit varying shrinkage values.

Several methods are available to measure polymerization shrinkage in composite resins. However, these methods vary due to differences in dimensional measurements (linear shrinkage and volumetric shrinkage) and types of shrinkage (total shrinkage and post-gel shrinkage). Linear shrinkage refers a two dimensional shrinkage changes, while volumetric shrinkage involves three- dimensional changes, allowing for the observation of multidimensional alterations.[9,10]

Total shrinkage refers to the measurement of all dimensional changes in the composite during polymerization, including both pre-gel and post- gel shrinkage. In contrast, post-gel shrinkage measures only the dimensional changes that occur after the gel point. Generally, current methods for measuring shrinkage often require specialized and costly equipment. Therefore, there is a need for a simpler method to determine volumetric shrinkage using readily available and affordable instruments, such as a digital microscope and ImageJ software, the latter of which can be downloaded free of charge.[4,10]

One approach to to addressing the limitations of composite resin is through the variations of filler materials, particularly by incorporating fibers. The use of fiber as fillers in composite resin offers several benefits, including the reduction of volumetric shrinkage, the ability to transmit loads within the matrix, and improvements in strength, stiffness, and fracture resistance. Fiber Reinforced Composite (FRC) is the term for composite resin with the use of fiber.[11]

Coconut (Cocos nucifera L./coir) is a versatile plant because every part of it offers a million benefits in human life. The part of the coconut plant that possesses high potential but has not been fully utilized is coconut fiber.[12,13] Coconut fiber (coir) is an innovation in natural fiber in composite resin production, and its application is being actively developed to optimize composite resins as promising materials for future dental restoration materials. Coir has a significant potential to replace synthetic reinforcing materials. However, fiber processing techniques must be carefully considered, as they play a critical role in determining the structure and properties of fibers used as reinforcing materials in dental composites. An important feature that affects the strength and toughness of a composite material is the bond strength between the filler material and the matrix.[14,15] This study focuses on the development of a low-viscosity Bis-GMA-based biocomposite resin for intermediate dental restorations.

Based on the above context, this study aimed to investigate the volumetric shrinkage of low viscosity Bis-GMA-based coir fiber composite resin as part of ongoing innovation in coir fiber composites development. The primary objectives were to assess the amount of volumetric shrinkage of coir fiber composite materials and to evaluate the shrinkage using scanning electron microscopy (SEM)

METHODS

This study used a true experimental design with a post-test-only control group. The synthesis of coir fibers and the fabrication of composite were conducted at the Electrochemistry and Corrosion Laboratory, Department of Chemical Engineering, Faculty of Industrial Technology and Systems Engineering, ITS. Volumetric shrinkage testing was performed at the ITS physical engineering laboratory, while SEM testing was conducted at BRIN Bandung. The materials used in this study included Bis-GMA (bisphenol A glycerolate dimethacrylate / 494356 Sigma–Aldrich), TEGDMA (triethylene glycol dimethacrylate / Sigma– Aldrich), DGEBA (diglycidyl ether of bisphenol A/ V-117-2207 Sigma–Aldrich), coconut fiber (obtained from Pasar Keputih Surabaya), camphorquinone (CQ; (Sigma–Aldrich Company St. Louis, MO, USA), and FiltekTM Z350 XT flowable restorative composite resin, which has a filler content of 65% and a primary composition of Bis-GMA, TEGDMA, and procrylat resins. Reagents used for alkali treatment included ethanol (C_2H_5OH; Merck), 4% w/w sodium hydroxide (NaOH; Merck), hydrogen peroxide (H_2O_2), urea (PT. Petrokimia Gresik), and demineralized water.

The materials for the marginal adaptation test included premolar teeth, Filtek Z250XT nanohybrid composite resin (3M ESPE, Germany), etchant gel (Universal Etchant 37% Phosphoric Acid Gel 3M ESPE, Germany), Universal Single Bond (3M ESPE, Germany), artificial saliva, and physiological solution (Widatra Bhakti, Indonesia). The tooth sample consisted of extracted maxillary first premolars with inclusion criteria requiring the teeth to be permanent maxillary premolar tooth with intact crown, no restoration, no caries, and no anomalies.

The treatment in this study involved several steps: the synthesis of coir fiber to obtain cellulose, the fabrication of composites, and testing. The synthesis of coir fiber began with drying fresh coconut fiber in an oven at 60℃ for 24 hours, followed by grinding it into powder. Subsequently, delignification was performed using 60% ethanol, and bleaching was conducted to remove the brown color of the coconut fiber using a mixture of H_2O_2, NaOH, and demineralized water in a rotary evaporator. Afterward, the fiber was dissolved using NaOH and urea. The dissolved fiber was then poured into a 99% ethanol antisolvent solution at a rate of 20mL/min and left to stand until solid cellulose sedimentation occurred. Next, solvent exchange was performed on the cellulose sediment using demineralized water until the pH reached neutrality. The sedimented fiber was then transferred to a freezer at -25℃ Subsequently, the frozen fiber was freeze-dried at -4℃ under a pressure of 5 mTorr for 24 hours. Finally, the coir cellulose fiber was ready to be used as a filler.[15]

Two types of composite samples were prepared in this study: composites with coir cellulose fiber fillers and composites with synthetic fillers as a comparison. The coir cellulose fiber composites were formulated with a composition of 70% coconut fiber by weight, 25% BisGMA resin by weight, and 5% TEGDMA by weight. The fibers and matrices were mixed for 60 minutes at 2℃ using a sonicator. Finally, the coupling agent DGEBA and camphorquinone were added, with DGEBA constituting 10% of the composite weight and camphorquinone 1% of the matrix weight, followed by mixing using a magnetic stirrer for 24 hours.[15]

For the volumetric shrinkage test, both composite samples were molded into an acrylic mold with a hole measuring 6mm in diameter 2 mm in thickness.[16] The mold was covered with a glass slide coated with Vaseline (Figure 1). Furthermore, the samples in the mold were then polymerized by irradiation using a light curing unit (Bluephase PowerCure Ivoclar Vivadent) for 20 seconds. Five samples were prepared for each group, with measurements taken at 0 minutes and 90 minutes post-irradiation.

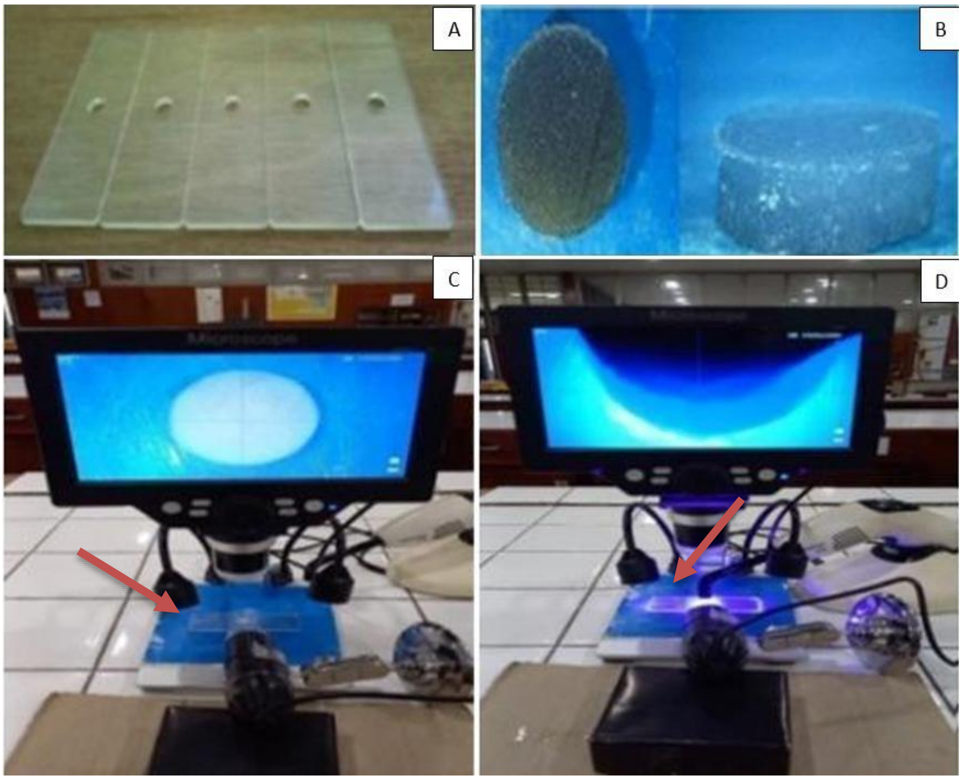

Figure 1. Volumetric shrinkage test (A) sample mold, (B) sample, (C) sample position on a digital microscope, (D) during polymerization

To measure volumetric shrinkage, images were captured using a digital microscope (Olympus CX-23) and analyzed with ImageJ v1.45 software to determine the projected area, assuming a constant thickness. The volumetric shrinkage is expressed as a percentage,calculated as the difference in volume before and after polymerization divided by the initial volume (before polymerization). This relationship is represented by equation (1), where εV denotes the volumetric shrinkage, ΔV represents the volume difference before and after irradiation or polymerization (that is $V-V_0$). The volumetric shrinkage data were analyzed using SPSS v26.

Marginal adaptation test was performed using Scanning Electron Microscope (SEM; Hitachi SU-3500). Sample preparation involved creating a class I cavity with a diameter of 3 mm and a depth of 4 mm on the occlusal surface (Figure 2). The cavity was rinsed with sterile distilled water using a 20 mL syringe and dried with cotton pellets. Next, 37% phosphoric acid gel was applied for 15 seconds for etching, followed by rinsing with water to remove debris. The teeth were then prepared for examination by applying a universal single bond agent for dentin bonding and irradiating with a light-curing unit according to the manufacturer's instructions. A flowable composite was inserted into an acrylic mold with a diameter of 6mm and a depth of 2mm from the cavity base and then cured. A packable composite is added to the cavity that has been filled with flow composite base until the entire cavity is filled and polished.

Furthermore, the specimens were soaked in artificial saliva for 24 hours at 37℃ in an incubator. After removal from the incubator, each specimen was sectioned into two parts in the bucco-palatal direction, parallel to the tooth axis, using a diamond disk. The sectioned teeth were then rinsed with 20cc of distilled water to clean the cut surface and dried at room temperature. The areas to be examined were marked with a colored marker and subsequently observed using SEM.

SEM observations were conducted on each tooth, focusing on the wall between the base of the flowable composite and the dentin to assess the presence or absence of gaps formed at magnifications of up to 100X. For each specimen, three microphotos were captured from three points along the interface: the right, the middle, and the left sections of the selected area with the largest width or distance.

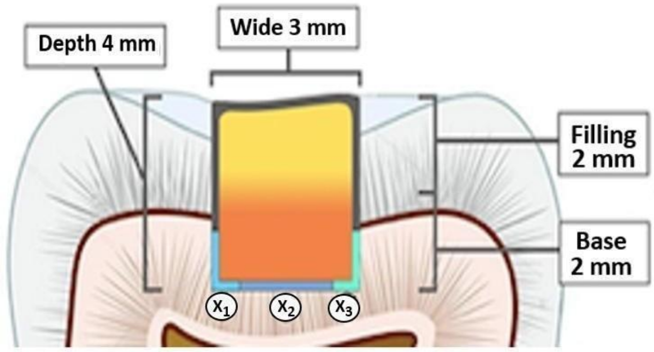

Figure 2. Sample preparation design for marginal adaptation test; X_1 is the largest gap on the left (sky blue), X_2 is the largest gap in the middle (blue), and X_3 is the largest gap on the right (green)

RESULTS

The minimum, maximum, median, mean, and standard deviation of the volumetric shrinkage test results for composites with coconut fiber (coir) and synthetic fillers, tested with a sample size of 5mm at 90 minutes after irradiation, are shown in Table 1.

Table 1. Volumetric shrinkage of flowable composite resin after 90 minutes (in %)

Type of filler	Min-max	Median	Average
Synthetic	0.0012-0.0149	0.0055	0.0401
Coconut fiber	0.0227-0.0808	0.0313	0.0064

Table 1 shows that the average volumetric shrinkage of composites with coconut fiber (coir) filler is significantly lower (0.0064%) compared to composites with synthetic fillers (0.0401%), with a value of more than 25 times. As a requirement for parametric testing, a normality test was performed before hypothesis testing using the Shapiro-Wilk normality test. The results, as illustrated in Table 2, indicate that the volumetric shrinkage in the coir fiber filler flowable composite resin group was normally distributed, with a significance value of 0.363 (sig > 0.05), whereas in the synthetic filler flowable composite resin group, it was not normally distributed (p = 0.005;sig < 0.05).

Table 2. Result of normality test using the Shapiro-Wilk test

Group	Sig. Shapiro-Wilk	Result
Composite with synthetic filler	0.005	Non-normal
Composites with coconut fiber (coir) filler	0.363	Normal

In addition to the normality test, a homogeneity test was conducted to determine whether the data in this study had equal variance or was homogeneous. The Levene test was used for this purpose, and the results (Table 2) demonstrated a significant value of 0.039 (sig <0.05), indicating that the data distribution was not homogeneous. Since the data were neither normally distributed nor homogeneous, a nonparametric test was used for hypothesis testing. This study used the Mann-Whitney nonparametric test, which resulted in s significance value of 0.047 (sig <0.05) (Table 3), indicating a significant difference in volumetric shrinkage between the coir filler flowable composite resin and the synthetic filler flowable composite resin.

Table 3. The results of Levene test and Mann Withney test of volumetric shrinkage on flowable composite with coir fiber filler and with synthetic filler

Test	Significance	Results
Levene	0.039	Not homogeneous
Mann-Whitney	0.047	Significant

The results of the marginal adaptation test (Figure 3) showed that the composite with coir fiber filler, when applied to premolar teeth, exhibited a smaller gap, with an average of 3.71 ± 2.43 μm wide. In contrast, the composite with synthetic filler formed an average gap of 10 ± 3.84 μm wide (Figure 4). These results confirm that the volumetric shrinkage in the composite with coir fiber filler is six times smaller than that of the composite with synthetic filler. Thus, the application of coir fiber as a flowable composite filler has proven to be successful, demonstrating superior performance in reducing volumetric shrinkage.

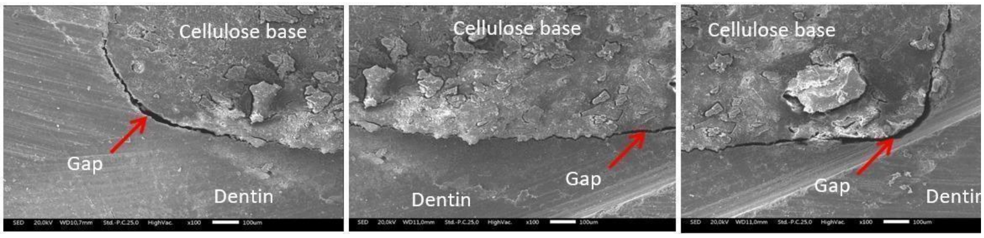

Figure 3. SEM results of the coir cellulose filler composite, the gap formed on the coconut fiber cellulose composite base with dentin walls, the average gap size is 3.71±2.43μm (100x magnification).

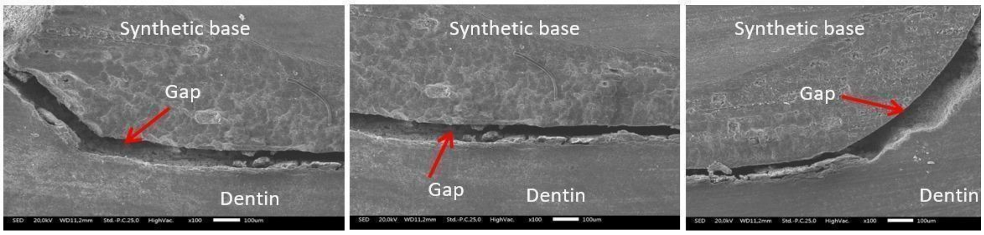

Figure 4. SEM test results of synthetic filler composite with gaps formed on the synthetic filler composite base with dentin walls. The average gap size is 10 ± 3.84 μm (100x magnification).

DISCUSSION

Coconut fiber consists of cellulose fiber, lignin, pyroligneous acid, gas, charcoal, tar, tannin, and potassium. In the fabrication of fiber composites, cellulose fiber serves as the primary reinforcement material. Lignin and other components must be removed because they are stiff and brittle.[13,14] Volumetric shrinkage was measured by quantifying the total dimensional changes in the composite during polymerization, including both pre-polymerization and post-polymerization shrinkage, collectively referred to as total shrinkage. A limitation of total shrinkage measurement is its lack of correlation with the shrinkage stress produced on the restored tooth structure.[15-18] However, total volumetric shrinkage provides valuable insights into the formation of three-dimensional polymer networks during the polymerization process. Therefore, total shrinkage is part of a comprehensive approach to studying the polymerization shrinkage of restorative composite materials.[8,16]

The test results indicated that volumetric shrinkage in synthetic filler flowable composite resin is significantly higher than that in coir fiber filler flowable composite resin. As shown in Table 1, the shrinkage of the composite with synthetic filler was more than six times greater than that of the coir fiber fille

The outcomes of the SEM test, which evaluated marginal adaptation (Figures 3 and 4), provided additional evidence that the volumetric shrinkage in the composite containing synthetic filler was six times larger than that of the composite containing coir fiber filler. The parameters in this study were based on research conducted by Tantbirojn et al. and Silva et al.[8,16] In Tantbirojn's study, volumetric shrinkage at 60 minutes ranged from 1.45 to 2.68%, while in Silva's study, it ranged from 1.18 to 3.45%. When compared with these previous research parameters, volumetric shrinkage in coir filler composites was significantly lower than in synthetic filler composites. Polymerization shrinkage is a well-known limitation of composite resin, as it compromises marginal adaptation, leading to marginal gaps and microleakage. Several factors that affect shrinkage include filler content, monomer molecular weight, and monomer conversion rate.[19-21] Additionally, the hollow fiber cross-section of coir, which resembles a sponge, can absorb heat, thereby slowing or preventing heat transfer, which may help mitigate shrinkage.

Fibers also help composites resist the tensile forces exerted from the margin toward the curing light, so it can maintain marginal adaptation by increasing resistance to dimensional changes and deformation during thermal loading. These findings are in line with the research of Schwendicke et al.,[22] who investigated the marginal integrity and mechanical properties of posterior composite restorations in vitro. Their study found that fiber-reinforced composite resins exhibited better margin quality and greater fracture resistance in molar teeth compared to particulate filler composites alone (Z250).[23] Findings from other in vitro studies by Garoushi et al. and Tsujimoto et al.,[24,25] support the conclusion that polymerization shrinkage can be reduced by incorporating fibers as fillers in composite resins. Their research demonstrated that fiber-reinforced composites exhibit lower shrinkage during polymerization and greater fracture toughness compared to conventional particulate composites resins.

Garoushi et al.[25] specifically evaluated the effect of short fiber-reinforced composite resins on polymerization shrinkage strain, shrinkage stress, and marginal microleakage. They reported that the presence of short fibers in composite resins increases resistance to microcracking, significantly reducing shrinkage stress and microleakage compared to restorations using particulate composite resins. This findings align with Tsujimoto et al.,[24] who reported that short-fiber reinforced composites exhibited much lower volumetric shrinkage (1.15%) compared to bulk-fill and conventional particulate composite resins, which showed shrinkage ranging from 1.3 to 2.4%.

The physical properties that affect marginal seal (marginal adaptation) originate from the restorative material itself and include the degree of attachment to the tooth structure, polymerization shrinkage, water absorption, solubility, and thermal expansion coefficient. Meanwhile, clinical factors that contribute to the failure of marginal seal formation include occlusal pressure, abrasion, and thermal stimulation.[26-29] The adhesion between the restorative material and the tooth structure is influenced by several factors, including operator skill, the properties of material used, the condition of the tooth structure, and the effectiveness of the adhesive. Additionally, key factors influencing microleakage include the coefficient of thermal expansion, polymerization shrinkage, restoration attachment, occlusal forces exerted on the cavity, and the filling technique.[27-30] Based on these results, low-viscosity coir fiber composites (flowable) show potential as intermediate layer (base) materials for coating the cavity walls. Their low elastic modulus allows them to reduce polymerization shrinkage by functioning as elastic artificial walls within the cavity.

These elastic walls counteract pressure resulting from polymerization shrinkage occlusal forces. In contrast, if the base material has a high elastic modulus, it will produce greater stress, as the material's elastic modulus increases during the polymerization reaction.[30] The primary limitation of this study is the assumption of uniform shrinkage when calculating volumetric shrinkage using the optical method based on projected surface area. This method also requires that the sample shape remain unchanged, which is true if the shrinkage is uniform and unrestricted. However, in flowable composite resins, sample slumping between the pre- and post- polymerization images may occur, requiring extra caution and careful handling.

Continuous real-time imaging of samples is achievable using optical method; however, manual determination of the projected surface area is time-consuming unless the measurement is automated. In addition, for certain samples, the boundary between the composite and the background may not be sufficiently distinct for the Wand tool in ImageJ to function effectively, so it is traced manually. In a pilot study, the variation in shrinkage values between the Wad tool and manual tracing was found to be less than 5%.

CONCLUSION

The volumetric shrinkage in composites with coir fiber filler (0.0064%) is significantly lower than that of composites with synthetic filler (0.0401%). These results, demonstrate that the application of coconut fiber as a filler in flowable composites is successful and yields superior performance in terms of volumetric shrinkage. The implications of these findings underscore the need for innovation in composite resin filler materials, shifting from synthetic fillers or synthetic fiber fillers to the use of natural fibers. Natural fiber fillers have the potential to address the limitations of current composite resin restorative materials, particularly in reducing volumetric shrinkage.

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