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Research Article

Effect of Exposure Time on the Microscopic Adhesion of Composite Nano Sisal

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Abstract

Composite resin has three main components: a resin matrix, filler particles (filler), and a coupling agent. The inorganic nature of composite resin filler particles contrasts with the organic components prevalent in tooth structure and some adhesives. Therefore, to increase adhesive, innovative composite materials containing organic fillers are required. The aim is to describe the effect of curing time (20, 30, and 40 seconds) on the microscopic adhesion strength of composite resins containing (a) nano sisal, (b) silane-treated hydroxyapatite nano sisal, and (c) Z350XT nanofiller (control) to dentin. This descriptive study investigated the potential of nano sisal, a sisal fiber-based filler in composite resin, as an alternative to conventional synthetic fillers. Samples were divided into three groups: nano sisal composite (Group A), nano sisal composite with a coupling agent (Group B), and Z350XT nanofiller composite (Group C). Premolar teeth were filled with all three composite groups and cured for 20, 30, and 40 seconds. Standardized samples were then extracted for microscopic adhesion observation via a Scanning Electron Microscope (SEM). SEM images of groups A, B, and C showed a gap in the adhesion between the composite resin and the tooth structure. The smallest adhesion distance in group A was at a curing time of 20 seconds (0.687 µm). Group B had a large adhesion distance at 20 seconds (15.747 µm). The smallest microleakege in group C was at 40 seconds of curing time. It was 0.644 µm. Microscopic examination using SEM reveals the presence of microscopic defects at the interface between the restoration and the tooth structure across all investigated curing times (20, 30, and 40 seconds).

Keywords: nano sisal; microscopic adhesion; composite resin; curing time

INTRODUCTION

Comprising a resin matrix, filler particles, and a coupling agent, composite resin fills both anterior and posterior teeth.[1] Additional components like activator-initiator, inhibitor, and optical modifier fine-tune its properties. Filler particles bolster the composite's strength by way of chemically binding the filler and the resin matrix,[2] while a crucial bond between the filler and matrix, facilitated by coupling agents, enhances its physical and mechanical properties.[3] A key challenge is polymerization shrinkage, which can form gaps between the filling and tooth, potentially compromising longevity.[4]

The infiltration of bacteria, food debris, and microleakege poses a significant threat to the restoration's longevity and success. This microscopic gap, often invisible to the naked eye, can lead to secondary caries, compromising the restoration's integrity and potentially necessitating further treatment.[5]

While traditional composite resins rely on inorganic filler particles for strength and wear resistance, their lack of inherent chemical adhesion to the organic tooth structure and adhesive materials presents a

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challenge.[6] This opens the opportunities to explore alternative fillers derived from natural sources, offering potential benefits in biocompatibility and sustainability. One such promising candidate is nano sisal, derived from readily available and inexpensive sisal fiber.[7] Through a series of processing steps, including alkalization, scouring, bleaching, and ultrasonication, sisal fiber is transformed into nanoscale cellulose whiskers. This organic nature eliminates the need for coupling agents, enabling seamless integration with the resin matrix and tooth structure.[8,9,10]

Achieving the perfect curing is crucial for successful composite restorations. Inadequate exposure leaves the bottom layer unpolymerized, compromising physical properties. Conversely, prolonged exposure intensifies shrinkage stress, potentially pulling the restoration away from the tooth and creating marginal gaps.[11]

Fifth-generation etch-and-rinse adhesives, a single-step primer and bonding system, facilitate strong attachment of composite resin to teeth. Etching with 30-40% phosphoric acid creates microscopic pores in enamel and dentin, providing entry points for adhesive infiltration. Primer and bonding fill these pores, establishing a micromechanical bond. Additionally, an adhesion promoter bridges the gap between the adhesive layer and the composite resin, forming a chemical bond crucial for long-term success.[12]

This study investigates the microleakege of microscopic nano sisal, nano sisal with a coupling agent, and Z350XT nanofiller composite resin to the tooth structure, analyzing exposure times of 20, 30, and 40 seconds.

MATERIALS AND METHODS

This descriptive study, conducted at the Molecular Medicine and Therapy Laboratory of FKIK UMY, investigated the microleakege of three composite resins to tooth structure: nano sisal composite resin (Group A), nano sisal composite resin with coupling agent (Group B), and Z350XT nanofiller composite resin (Group C, control). Caries-free permanent premolars with class V GV Black cavities served as sample teeth.

Nano sisal preparation

Nano sisal preparation began with cutting sisal fiber to a smaller size. The prepared fiber was then subjected to a three-step alkaline treatment: soaking in a 4% NaOH solution at 80°C for 2 hours with continuous magnetic stirring, followed by filtration and thorough rinsing with water to remove residual alkali.

Following the alkaline treatment, the sisal fiber underwent a four-stage bleaching process. In each stage, it was immersed in a solution of 27 g NaOH, H_2O_2, and liquid chlorite at 80°C for 4 hours with continuous magnetic stirring. After each immersion, the fiber was filtered and washed with distilled water. Finally, it was dried in an oven at 60°C for 24 hours.

The dried fiber was pulverized into a fine powder and subsequently subjected to acid hydrolysis with 65 wt% sulfuric acid at 50°C for 50 minutes under constant stirring. This reaction was quenched by rapid dilution with ice, and the resulting suspension was centrifuged at 10°C and 5000 rpm for 30 minutes to remove unreacted components. Subsequent dialysis against distilled water effectively eliminated free acids from the dispersion. Finally, ultrasonication using a Cole Palmer Ultrasonic Processor facilitated the formation of nano-whiskers. Filtration steps removed any remaining aggregates, and the final nano sisal dispersion was freeze-dried to obtain its semi-solid form.

Preparation of nano sisal composite samples

Nine caries-free permanent premolars were prepared with kidney-shaped Class V G.V. Black cavities approximately 1–1.5 mm deep using round, fissure, and inverted cone burs. The teeth were then divided into three groups and cleaned with a cavity cleanser. An acid etching procedure with Scotchbond Etchant 3M ESPE for 20 seconds was followed by the application of Adper Single Bond 2 bonding. The bonding material was air-dried and light-cured for 20 seconds.

For Group A, 0.0696 g of semi-solid nano sisal was weighed and mixed with 0.09186 g Bis-GMA, 0.04017 g TEGDMA, 0.00094 g UDMA, and 0.00094 g Camphorquinone on a glass plate to create the nano sisal composite resin. Group B followed the same procedure as Group A but with the addition of a coupling agent. Group C premolars were filled with Z350XT nanofiller composite resin (3M ESPE). All samples (Groups A, B, and C) were then exposed to their respective composite resins for 20, 30, and 40 seconds at a light curing unit distance equivalent to the thickness of a celluloid strip.

Scanning Electron Microscope (SEM) Test

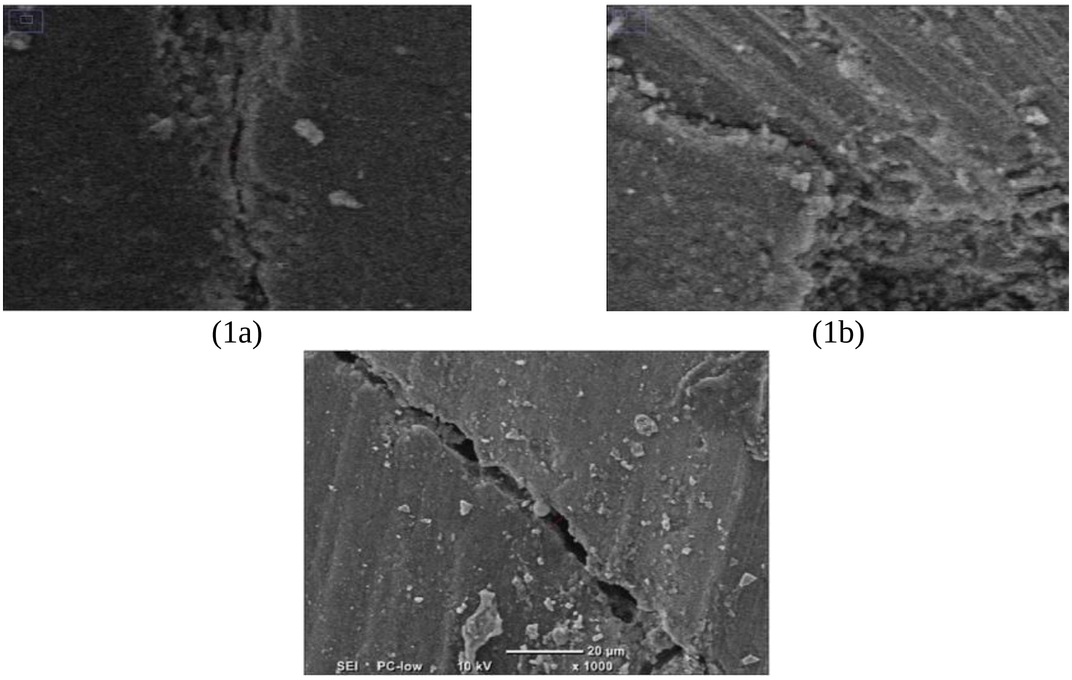
For the Scanning Electron Microscope (SEM) analysis, filled premolar tooth samples were sectioned into cubic specimens measuring 1 cm per side. Gap measurements were then performed at three designated points on each specimen to calculate the average microleakege.

RESULT

Table 1. Mean results and standard deviation of microleakege for nano sisal composite resin, nano sisal composite resin with a coupling agent, and Z350XT nano filler composite resin

	Mean of Microleakege (μm)		
	Nano Sisal (A)	Nano Sisal Coupling Agent (B)	Nanofiller Z350XT (C)
	Mean ± SD	Mean ± SD	Mean ± SD
20 s	0.687 ± 0.064	15.747 ± 0.508	2.636 ± 0.017
30 s	0.820 ± 0.046	1.827 ± 0.041	0.953 ± 0.325
40 s	5.437 ± 0.949	1.414 ± 0.342	0.644 ± 0.074

Microleakege was measured at three points per sample to calculate the average for each group (Table 1). Microscopic gaps were observed in all samples. Descriptive analysis revealed the highest average microleakage (15.747 µm) in the nano sisal with coupling agent group at 20 seconds exposure. Conversely, the Z350XT nanofiller group at 40 seconds exhibited the smallest distance (0.644 µm). Microscopic images of nano sisal composite resin adhesion at 20, 30, and 40 seconds exposure time are shown below.

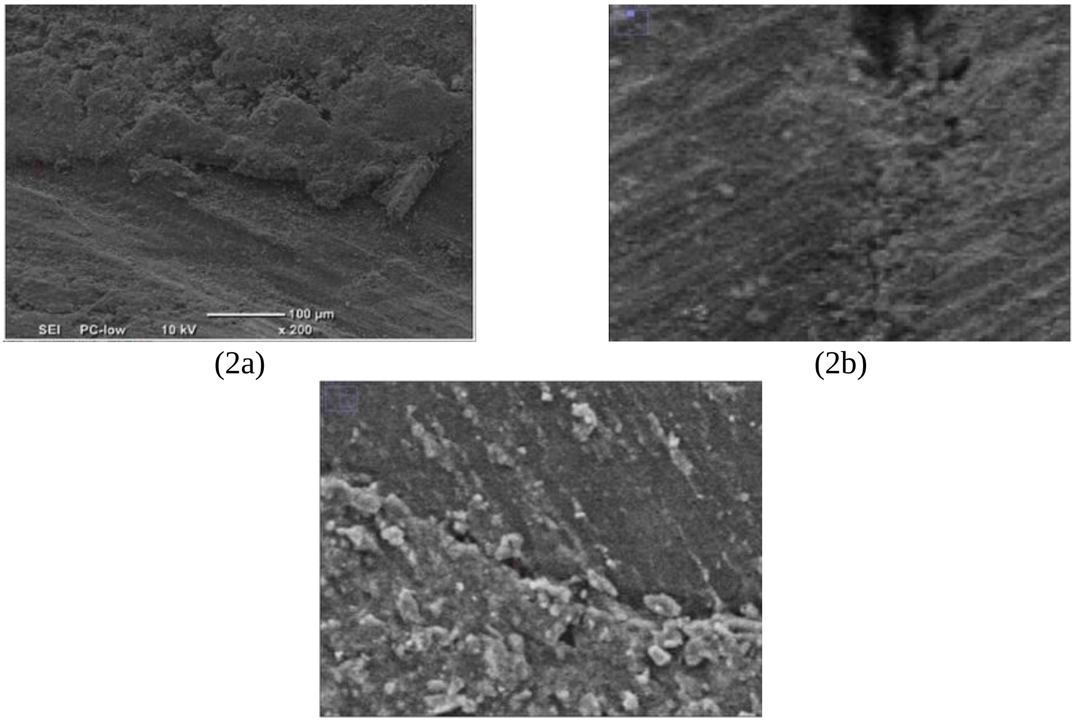


(1a) (1b)

(1c)

Figure 1. Image of microleakege of nano sisal composite resin and tooth structure with exposure time (1a) 20 seconds; (1b) 30 seconds; (1c) 40 seconds (1000x magnification)

Figure 1 describes the microleakege of nano sisal composite resin to tooth structure at various exposure times: (a) 20 seconds, (b) 30 seconds, and (c) 40 seconds. Notably, all images reveal microleakege, creating adhesion gaps at the interface. Image (a), with 20 seconds of exposure, exhibits the smallest microleakege (0.687 µm), followed by image (b) at 30 seconds (0.820 µm). However, image (c) with 40 seconds of exposure shows a significantly larger gap, with a microleakege of 5.437 µm, evident in the prominent void space under the SEM image. Additionally, the microleakege of nano sisal with a coupling agent and varying exposure times (20, 30, and 40 seconds) is described below.

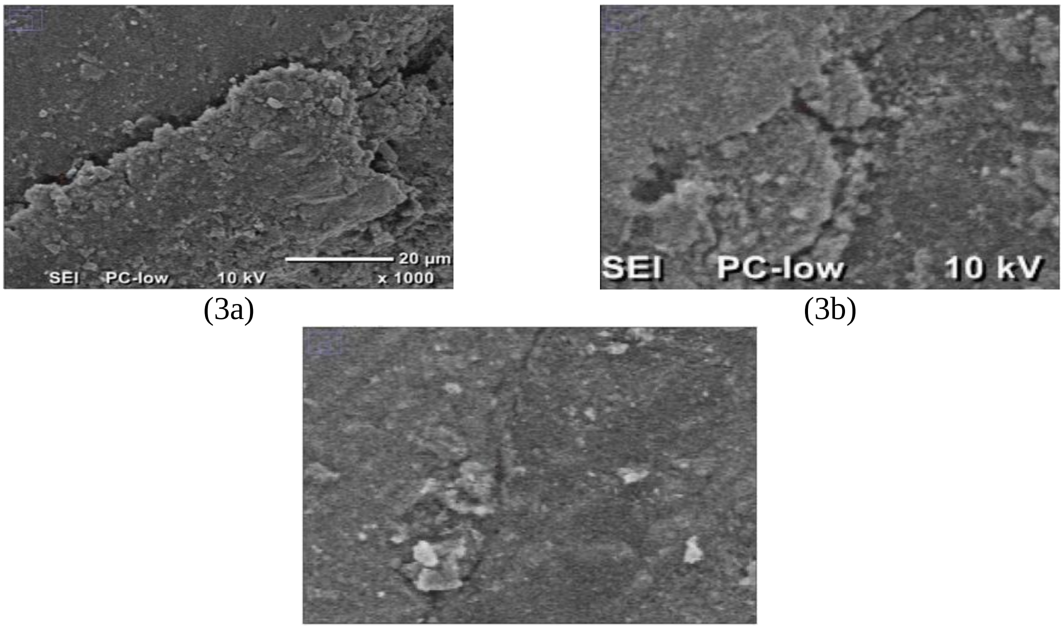


(2a) (2b)

(2c)

Figure 2. Microscopic view of the attachment of nano sisal composite resin with a coupling agent and tooth structure with exposure time (2a) 20 seconds; (2b) 30 seconds; (2c) 40 seconds (1000x magnification)

Arrows in all images pinpoint gaps at the restoration-tooth interface. The 20-second coupling agent group (a) exhibits the highest microleakege (15.747 µm), significantly exceeding those at 30 seconds (image b, 1.827 µm) and 40 seconds (image c, 1.414 µm). The microleakege of the Z30XT nanofiller composite resin with varying exposure times (20, 30, and 40 seconds) is presented below.



(3a) (3b)

(3c)

Figure 3. Image of microleakege of Z350 XT nanofiller composite resin and tooth structure with exposure time (3a) 20 seconds; (3b) 30 seconds; (3c) 40 seconds (1000x magnification)

For the Z30XT nanofiller group (Figure 3), the 20-second exposure sample (image a) exhibits the highest microleakege (2.636 µm), followed by a decreasing trend at 30 seconds (image b, 0.953 µm). Notably, the Z350XT nanofiller group at 40 seconds (image c) shows the smallest average distance (0.644 µm), suggesting potentially superior adhesion based on its compact appearance in the SEM image. However, further analysis beyond morphology, such as microleakege assessment and mechanical testing, is necessary to definitively conclude the adhesive performance of Z350XT at 40 seconds compared to other groups.

DISCUSSION

This study investigated the microleakege of nano sisal composite resin, nano sisal composite resin with a coupling agent, and Z350XT nanofiller composite resin to tooth structure at 20, 30, and 40 seconds LED curing unit exposure time. Microleakege images obtained via Scanning Electron Microscope (SEM) were subsequently analyzed using an image processing application to measure attachment distances.

The predominant mechanism of attaching nano sisal composite resin, nano sisal with a coupling agent, and Z350XT nanofiller to teeth is often perceived as a simple replacement of tooth's inorganic hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$ with synthetic resin.[2] This primarily occurs through the formation of resin microtags on the enamel surface induced by acid etching. This process transforms the smooth enamel into an irregular surface with increased surface-free energy. Consequently, when an adhesive encounters this roughened enamel, its resin component penetrates the surface through capillary action. Finally, the resin monomers polymerize, solidifying the material's bond with the enamel.[4]

Following acid etching, a primer solution containing hydrophilic monomers dissolved in acetone, ethanol, or water is applied to maintain collagen tissue hydration and remove residual water. This removal is crucial for optimal infiltration by the bonding agent, which contains hydrophobic monomers alongside hydrophilic components. The hydrophilic portions of the bonding agent diffuse into the dentin, forming a hybrid layer and resin tags for micromechanical retention. The success of hybrid layer formation hinges on the combined ability of the primer and bonding agent to penetrate the dentin surface effectively.[2,12]

Four primary mechanisms govern the attachment of composite resin materials to tooth structure: mechanical, adsorption, diffusion, and a combination of these. The mechanical mechanism involves resin penetration into the tooth surface, forming resin tags for micromechanical retention. The adsorption mechanism, on the other hand, relies on chemical bonding with either inorganic components like hydroxyapatite or organic components like collagen type I present in the tooth. The diffusion mechanism involves the deposition of substances on the tooth surface, enabling subsequent resin monomer attachment through mechanical or chemical interactions. Finally, the combination mechanism synergistically employs elements of all three aforementioned mechanisms.[4]

SEM analysis of nano sisal, nano sisal with coupling agent, and Z350XT groups revealed microscopic gaps between the restoration and tooth structure, with varying attachment distances. These gaps, indicative of potential adhesion loss, can arise from polymerization shrinkage within the composite resin. Notably, the resin matrix, composed of monomers with carbon double bonds, is held together by weak Van der Waals forces before polymerization. During polymerization, these monomers covalently bond to form a polymer, shortening the individual monomer chain bond lengths from ~0.3–0.4 nm to ~0.15 nm. This dramatic contraction, termed polymerization shrinkage, leads to a net decrease in composite resin volume and the observed micromechanical gaps.[13–15]

Nano Sisal composite resin, an organic-based restorative material, exhibits three primary bonding mechanisms: mechanical, electrostatic, and chemical. Mechanical bonding involves physical interlocking between the resin and the substrate surface, often facilitated by microscopic irregularities. Electrostatic bonds arise from attractive forces between charged groups within the resin and the substrate, such as ionic and Van der Waals interactions. Lastly, chemical bonding involves covalent or ionic linkages formed between the resin and the substrate, potentially mediated by silane coupling agents for enhanced interfacial adhesion.[16]

Nano sisal composite resin showed varying microleakege at different exposure times: 0.687 µm at 20 seconds, 0.820 µm at 30 seconds, and 5.437 µm at 40 seconds. Notably, the 20-second group exhibited the smallest distance compared to the longer exposure groups. This trend aligns with research by Dhamayanti et al. (2014), suggesting that longer exposure durations induce a higher degree of conversion – the percentage of double bonds converted into single bonds during polymerization.[17,18] While a high degree of conversion strengthens the composite, it can also increase polymerization shrinkage, potentially leading to the observed gap between the resin and tooth structure.[2] Conversely, lower conversion, as with the 20-second group, could decrease polymer bonding and shrinkage, possibly explaining the smaller attachment distance.[15] The microleakege of nano sisal with an irradiation time of 20 seconds is 0.687µm.

The introduction of a coupling agent significantly affected nano sisal adhesion distance. At 20 seconds, the coupling agent group exhibited a substantial increase, reaching 15.747 µm compared to the uncoupled nano sisal composite resin. Interestingly, this trend reversed at longer exposure times, with subsequent decreases down to 1.827 µm at 30 seconds and 1.414 µm at 40 seconds. This complex behavior may be influenced by two opposing factors. On the one hand, coupling agents generally reduce shrinkage stress by providing additional bonding sites between the filler and matrix. The absence of a coupling agent allows for stress release through internal cavities within the filler-matrix interface, contributing to lower shrinkage stress but potentially compromising mechanical properties.[15] On the other hand, the specific coupling agent used in this nano sisal (DGEBA) possesses inherent stiffness, which, according to previous research, can elevate shrinkage stress during polymerization. This increased stress could explain the larger gap observed in the SEM image at 20 seconds for the coupling agent group.[18]

Among all groups, the nano sisal with coupling agent and 20-second exposure exhibited the highest attachment distance. This seemingly advantageous finding might be attributed to incomplete light penetration, potentially reducing free radical formation for ideal polymerization.[19] Inadequate polymerization, as evidenced by microleaks, can jeopardize the restoration's strength.[20] Notably, this study used a bulk placement technique, where the composite resin is applied all at once. This approach could have hindered light transmission to the bottom layer, leading to incomplete polymerization at the interface with the tooth structure.[21]

With a microleakege of 0.644 µm at 40 seconds, the Z350XT nanofiller exhibited the smallest gap among all nano sisal and Z350XT groups analyzed (both with and without coupling agents) and at all exposure times (20 and 30 seconds). This superior adhesion, evident in the SEM image as well, may be attributed to the specific resin composition of Z350XT. Notably, it contains TEGDMA, a low-molecular-weight, low-viscosity monomer known to enhance resin mobility. This increased mobility could facilitate deeper light penetration and complete polymerization, potentially mitigating shrinkage stress and promoting stronger adherence to the tooth structure. UDMA and Bis-EMA, also present in Z350XT, offer higher molecular weights for improved mechanical properties, while TEGDMA and PEGDMA act as viscosity adjusters. Further investigation, perhaps comparing specific resin formulations and their influence on polymerization depth and adhesion, could provide valuable insights into optimizing composite resin performance.[22]

Z350XT nanofiller utilizes PEGDMA resin in its matrix to partially substitute TEGDMA, aiming to mitigate the polymerization shrinkage inherent to this restoration material.[22] This aligns with studies like Lin et al. (2020), demonstrating that resin matrix composition significantly impacts shrinkage. Notably, their research found that replacing most TEGDMA with PEGDMA in Z350XT substantially reduced shrinkage.[23]

Beyond influencing mechanical properties and wear resistance, filler loading also critically impacts polymerization shrinkage in composite resins. Z350XT nanofiller exemplifies this, featuring a high inorganic filler content (78.5%) compared to nano sisal (60%).[22] This elevated level, achieved through a combination of non-agglomerated zirconia (4-11 nm), non-agglomerated silica (20 nm), and zirconia/silica nanoclusters (20 nm), demonstrably minimizes shrinkage. Indeed, studies like Gonçalves et al. (2015) highlight that increasing filler load reduces the resin matrix volume and, consequently, the overall shrinkage. This effect stems from a decreased concentration of carbon double bonds in the resin, minimizing the potential for contraction during polymerization.[25]

CONCLUSION

The present study revealed diverse adhesion profiles among the analyzed materials. Notably, nano sisal with a coupling agent (Group B) exhibited the largest microleakege at a surprisingly short exposure time of 20 seconds, while Z350XT nanofiller (Group C) displayed the smallest gap at 40 seconds, and nano sisal composite (Group A) described the smallest microleakege at 20 seconds. Nano sisal composite has better properties of microleakege when curing exposed at 20 seconds. These findings suggest a complex interplay between exposure time, coupling agent presence, and material composition influencing bond strength.

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