



Effect of Nanosilica Addition on Microshear Bond Strength Self Adhesive Composite

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ABSTRACT

Introduction: The flowable composite resin is self-adhering as it includes an adhesive. These substances have been proposed to remove the requirement for a separate bonding administration phase; hence it may be thought to begin the eighth generation of dental adhesive systems.

Objectives: Study the effect of nanosilica addition on shear bond strength (SBS) of two self-adhesive resin composites. The main disadvantage of flowable dental composite resin is its lower strength compared to traditional composite resin.

Materials and methods: In this study, we used 40non-carious sound human molars, recently extracted, Vertise Flow, Fusio Liquid Dentin, and Vertise Flow modified with nano silica filler. Fusio Liquid Dentin is modified with Nanosilica filler. Then, the samples were placed in distilled water at room temperature; that was changed every three days for the specimens and aged for 3 months. Micro SBS test were applied.

Results: Fusio Control (17.31±2.66 MPa) revealed the lowest mean μ-SBS followed by Fusio+Nanosilica (19.08±1.45 MPa), Vertise Control (21.52±1.15 MPa), then Vertise+Nanosilica (23.53±1.56 MPa) at p≤0.001 after 24 hours .Fusio Control (12.83±1.88 MPa) revealed the lowest mean μ-SBS, followed by Fusio+Nanosilica (16.99±2.01 MPa), Vertise Control(18.8±1.47 MPa), then Vertise+Nanosilica (19.91±1.33 MPa) at p≤0.001 with an insignificant difference between Vertise control and Vertise+Nanosilica after 3 months.

Conclusions: Nanosilica particles addition to the self-adhesive materials used in this study might have a positive effect on SBS.

KEYWORDS: Nanosilica, self-adhesive, μ -Shear Bond Strength, Flowable Composite.

1. INTRODUCTION

Flowable composites are distinguished by their fluidity. The amount of fluidity varies significantly from one product to another [1]. In order to make the mixture less viscous, the filler quantity was reduced by 20-25%, resulting in flowable composites. Consequently, these substances have a lower modulus of elasticity and less rigidity compared to traditional hybrid composites, yet they exhibit higher stress due to polymerization shrinkage and volumetric shrinkage [2]. This material is being considered for use as a stress-absorbing layer, a bonding agent for orthodontic brackets, and a restorative material for small-sized class I cavity and fissure sealants when combined with hybrid composite resin. They should only be used in places that are not subject to direct stress [3].





One benefit of flowable resin composite is its excellent characteristics. That material's fluidity feature is enhanced, making its installation easier and facilitating its adaptation to cavity walls [4]. The polymer chains have less relative mobility during polymerization, which results in increased stress, and this helps to mitigate the negative impact of polymerization shrinkage, which is more common in stiffer materials [5]. This is why the suggestion for softer resin has been made. A dental bonding system must be used in conjunction with flowable composite resins since these resins do not possess adhesive characteristics on their own. A novel class of restorative materials called "self-adhering composite resins" was recently introduced, which is based on a leading-edge resin-based material that combines flowability and self-adhesion capabilities [6]. The flowable composite resin incorporates an adhesive resin and is self-adhering. An effective bonding method for self-adhering flowable composite resin involves etching dentin and enamel with a functional monomer like gycerophosphate dimethacrylate (GPDM) [7]. As an additional functional monomer, it includes hydroxyethyl methacrylate (HEMA), which is used extensively in dental adhesives to increase dentin wetting and resin infiltration. It is said that these materials streamline the direct restoration process by avoiding the necessity for a separate phase for bonding application. Due to this, it might be seen as the beginning of the eighth generation of dental adhesive systems or as a connection between all-in-one adhesives and flowable composite resins. Flowable composites' mechanical properties, such as diametral tensile strength, fracture toughness, and compressive strength, usually vary between 60% and 90% of those of standard composites [7].

Additionally, compared to traditional composites, particularly those that are heavily packed, the wear resistance is significantly lower [1]. Since flowable dental composite resin requires a small quantity of filler to achieve its ease of handling and low viscosity, its strength is lower than that of traditional composite resin. This is the fundamental downside of this material compared to conventional resin [8]. The addition of nanoparticles can enhance their mechanical qualities. Nanoparticles are one-of-a-kind due to their tiny size, enormous surface area, high surface energy, and high atomic proportions on their surface. Composite resin often contains particles of silica and titanium dioxide, which were added to enhance mechanical qualities, such as bond strength and flexural strength [9]. Due to its excellent biocompatibility [10], superior aesthetics, and general usage as a filler in dentistry, silica nanoparticles were selected for reinforcement of the resin composite in this investigation. This study aimed to investigate how incorporating nanosilica particles into a common commercially available self-adhesive flowable dental composite resin affected its microshear bond strength.

2. METHODOLOGY

2.1. Materials

The materials employed in this study included a number of materials: first, **Fusio Liquid Dentin**, a self-adhesive flowable composite; second, **Vertise Flow**, a self-adhering flowable composite that has an average particle size of 1 micron, and includes activators, stabilizers, and colourants; and lastly, **Nano silica**, which was provided as a quality control unit with a certificate of analysis (COA) (See Table 1).

2.2. Methods

2.2.1 Ethical Consideration:

This study was performed after the approval of the Institutional Review Board (IRB) of Sinai University (SU.REC.2025 (53 H)).





2.2.2 Sample Size Calculation

The sample size was calculated using the PASS 15 software (NCSS LLC, TX, USA) with a one-sample t-test procedure. The computation used an alpha of 0.05 and a target power of 80%. As a consequence, 40 samples were used in the investigation, with 20 assigned to each category.

Table 1. List of the utilized materials

Material	Components	Manufacture			
Fusio Liquid Dentin	UDMA, TEGDMA, HEMA, META, nano-sized amorphous silica, silane-treated barium glass, minor additives, and a photo-curing system	Pentron Clinical (Orange, CA, USA), with batch number 4993575 and reference number N2188			
Vertise Flow	uncured methacrylate ester monomers (18–40%), four types of inert mineral fillers, including a prepolymerized filler, one-micron barium glass filler, nano-sized colloidal silica, and nano-sized ytterbium fluoride	Kerr Corporation with batch number 5079373 and reference number 34402			
Nano silica	white powder and was used in a quantity of 5 grams. It was dispersible in water or ethanol, with an average particle size of over 50 nm as measured by TEM.	NanoTech Egypt Company			

2.2.3 Selection of Teeth

The research used 40 healthy (human) upper and lower teeth that were free of caries. A scaler was used to remove any remaining soft tissue from the roots. A 7X magnification lens was used to inspect the teeth to rule out any teeth that could have cracks. After rinsing with water, the teeth were preserved in distilled water at 4°C for no more than one month before testing [11–13]. Each tooth's acrylic resin block had dimensions of 1.5 cm X 1.5 cm X 2 cm, and the cement-enamel junction of the crown was flush with its top surface. Each tooth's occlusal dentin surface, which is located superficially (0.5 mm below the dentino-enamel junction, or DEJ) was exposed. A cylindrical flat-ended diamond stone (ISO #111/012) was utilized to carve two guide grooves into the distal and mesial surfaces, allowing for the determination of the DEJ level. After that, a high-speed handpiece and a generous amount of air-water spray were used to smooth out these grooves. A line was drawn on the proximal surfaces using a lead black pencil, parallel to the DEJ but 0.5 mm below it. A circle representing the surface level was then methodically formed around the tooth by joining the two lines on the proximal surfaces using the lingual and buccal surfaces. The tooth's enamel and dentin structure above the line was ground flat using a cylindrical flat-ended diamond stone to expose the superficial dentin level. Slightly below the dental enamel-dental junction (DENJ), perpendicular to the tooth's longitudinal axis, and parallel to the occlusal surface was the level of the superficial dentin.

2.2.4 Specimens Grouping

Two equal groups of specimens were randomly assigned based on the type of self-adhesive flowable composite that was utilized. The ten participants in each group were split into 2 equal subgroups, one for the control group consisting of an unaltered self-adhesive flowable composite, and the other for the group that had its composition altered by adding 0.05% silica nanoparticles. Depending on whether the samples were stored for 24 hours or three months, each subgroup was split into two classes of five samples.





2.2.5 Synthesis of Silica Nanoparticles

The nanosilica particles were supplied by NanoTech Egypt Company. To create uniformly sized silica nanoparticles that were both monodisperse and ultrasonicated, they employed a sequential approach based on the sol-gel technique. Trans-esterified orthosilicate (TEOS) was hydrolyzed in an ethanolic environment to produce the silica particles [13]. The formation of monodisperse and spherical silica nanoparticles was initially documented by Stöber et al. [14]. They used aqueous alcohol solutions of silicon alkoxides with ammonia as a catalyst to create nanoparticles of varying sizes, with a narrow size distribution (50 nm - 1 μ m). Particle size is alcohol and silicon alkoxide-type dependent [15,16].

Quantity and Form: Transmission electron microscope (TEM) images were captured using an accelerating voltage of 200 kV on a JEOL JEM-2100 high-resolution TEM (Fig. 1).

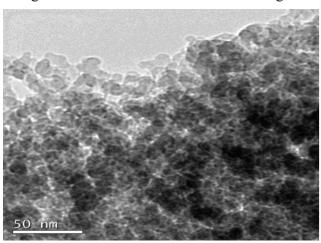


Fig. 1. TEM photomicrograph of the morphological shape of the synthesized nanosilica

2.2.6 Modification of Flowable Composite

The MÜller S1J-180 precision drill/grinder was used for mixing, along with a Dentsply lentulo spiral-paste carrier #4. A 2 ml predarkened syringe tube was used to submerge the lentulo spiral in a combination of composite resin and nanosilica. A syringe plunger was fitted into the syringe tube, the drill/grinder with the lentulo connected was removed after one hour of mixing, and the mixture was injected into elastomer molds. An LED light curing equipment (Radii Plus 8-mm tip, SDI Limited, Bayswater, Victoria 3153, Australia) was used to cure the samples. A light guide was kept 2 mm from the material surface and held perpendicularly for all photopolymerizing processes. During the research, the curing unit's light output was confirmed by an integrated radiometer, and the high-power curing mode (HIP) was consistently used. The curing light had an intensity of 1,500 mW/cm², and the samples were exposed for 20 seconds (disc samples) or 20 seconds plus 20 seconds (square samples). The samples were shaped and measured using 1500- grit SiC paper after hardening [17].

2.2.7 Micro-Shear Test Sample Preparation

A PVC (polyvinyl chloride) tube with an internal diameter of 25 mm was used to hold each material slice while the specimens were being made and tested. The tube had previously been filled with acrylic resin. As a mold for the flowable composite, we utilized a 2 mm long and 1 mm inside diameter polyethylene tube (TYG-030, Saint-Gobain Performance Plastic,

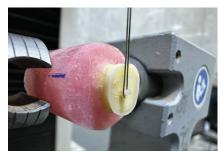


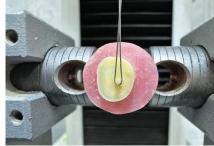


Miami Lakes, FL, USA). On top of the dentin substrate, four slit tubes were spaced out. Flowable composite was injected into each tube while stabilizing devices were used to keep them in place. Following the manufacturer's instructions, the flowable resin composite was carefully inserted into the silicon microtubules using the micro-applicator nozzle. The dentin surface was prepared by bonding resin composite cylinders to it, and the polyethylene tubes were cut and removed employing a sharp lancet No. 15. After that, for the next three months, not all the specimens, only the 3 aged classes (20 teeth) were submerged in room-temperature distilled water, which was replaced every three days [7, 11].

2.2.8 Micro-Shear Bond Strength (SBS) Test

Specimens having air bubbles or gaps at the interface were discarded after being examined with a light stereomicroscope (Nikon MA100, Japan) at 30× magnification prior to testing. Connected to the lower fixed head of the universal testing machine (Instron model 3345, England) was the acrylic block containing the specimen. A 0.14-inch-diameter stainless steel wire was fastened to the testing machine's upper moveable head and positioned as close to the composite/enamel or dentin contact as feasible for each tube to undergo a μ SBS test. Tensile force is delivered up until specimen failure at a crosshead speed of 1.0 mm/min. The machine software, BlueHill 3 Instron England, calculated the SBS in MPa by dividing the force needed for failure (Newton) by the surface area (mm²) (See Fig. 2). For μ -SBS calculations, the load at failure was divided by the bonding area to express the bond strength in MPa: $\tau = P/\pi r^2$, where $\tau =$ bond strength (in MPa), P load at failure (in N) $\pi =$ 3.14, r radius of microcylinder (in mm) [7].





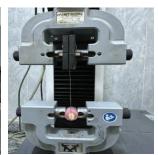


Fig. 2: Micro-shear bond strength test.

2.2.9 Test Disposal of Extracted Teeth

After usage, extracted teeth and their remnants are collected in a hazardous waste container and sent to be incinerated.

2.2.10 Statistical Analysis

Results are shown as means with standard deviations (SD). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to examine the data for normality. The normal distribution of $\mu\textsc{-}SBS$ was observed, and the influence of various restorative materials on the mean $\mu\textsc{-}SBS$ throughout each assessment period was studied using multivariate ANOVA. The influence of various follow-up durations on the mean $\mu\textsc{-}Shear$ Bond Strength (MPa) within each restorative material was investigated using an independent t-test. P < 0.05 was used as the significance level. Statistical analysis was conducted employing IBM® SPSS® Statistics Version 22 for Windows, developed by SPSS Inc. and owned by IBM Corporation in New York, USA.



3. RESULTS

Effect of Different Restorative Materials

1. Effect of Different Tested Materials after 24-Hour Testing Period on the Mean μ-SBS

Vertise containing nanosilica showed the highest bond strength value (23.53 ± 1.56) with a significant difference from the Fusio control that showed (17.31 ± 2.66) . Comparing Fusio with and without Nanosilica, a significant difference was noted as Fusio containing nanosilica showed the highest mean value. Similarly, Vertise containing nanosilica showed the highest mean value with a significant difference from Vertise control at p \leq 0.001 (See Table 2).

Table 2: Mean and standard deviation (SD) of l for different resin composite system and relative E:D thickness.

		Fusio				Vertise				p-value	
		Control		Nanosilica		Control		Nanosilica			
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	•	
μ-shear Bond	24 hrs	17.31a	2.66	19.08 ^b	1.45	21.52°	1.15	23.53 ^d	1.56	≤0.001*	
Strength (MPa)	3 Months	12.83 ^a	1.88	16.99 ^b	2.01	18.80°	1.47	19.91°	1.33	≤0.001*	
p-value		≤0.001* 0.014*		≤0.001*		≤0.001*					

Means with the same letter within each row are not significantly different at p=0.05.

2. Effect of Different Tested Materials after 3-Month Testing Period on the Mean μ -Shear Bond Strength (MPa)

Table (2) and Figs (3 & 4) display the means \pm SD for μ -SBS for various restorative materials and follow-up durations. Across all samples evaluated, the nanosilica-modified samples had μ -shear bond strengths that were greater than the unmodified control sample. The Vertise samples that were treated with nanosilica had the greatest μ -shear bond Strength (MPa), while the unaltered samples exhibited a diminishing trend. It is noticeable that the standard deviation rises with the addition of nanosilica. The findings demonstrated that the μ -shear bond strength (MPa) was significantly larger in the data collected after 24 hours compared to the data collected three months later.

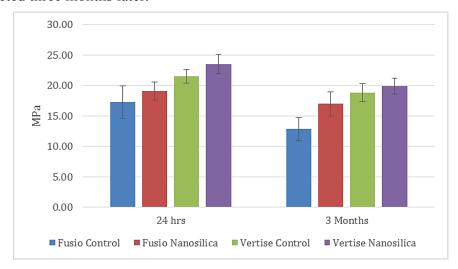


Fig. 3: Histogram showing the mean μ-shear Bond Strength (MPa) for different restorative material within each follow-up periods

^{*=} Significant





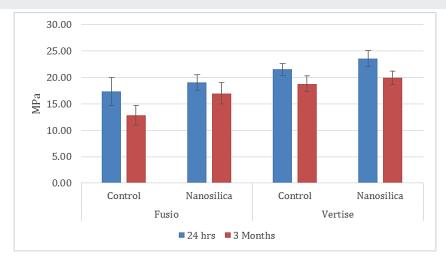


Fig. 4: Histogram showing the mean μ -shear Bond Strength (MPa) for different follow-up periods within each restorative material.

4. DISCUSSION

The most notable property of silica particles, which often appear as a fine, white, amorphous powder or colloid solution, is their very high surface area and nonporous, smooth surface. These properties enable them to provide strong physical contact when placed in a polymer matrix. You may find silica particles in diameters ranging from nanometers to micrometers on the marketplace. The mechanical characteristics of silica/polymer composites are greatly affected by the dispersion of silica fillers, by extension, and by how well the polymer and silica are compatible with one another. Given that the majority of polymers are hydrophobic, it is crucial to modify the silica surface in order to increase its dispersion in the matrix and enhance the interfacial contact between the two. Physical and chemical approaches both work well to alter the surface of silica fillers [18].

Flowable self-adhesive resin composites have been the subject of little research into how adding nanosilica affects their microshear binding strength to dentin and how aging (after three months) affects this bond strength. In addition to being more convenient and quicker, in vitro testing can reveal which elements are most harmful to the efficiency of bonds over the long run. With the addition of nanosilica, all mechanical characteristics were shown to be improved. This improvement made it more resistant to wear, flexure, and fractures. The rheological characteristics were further enhanced by the use of silica nanofibers [19].

Before adding nanosilica to become Fusio + nanosilica and Vertise Flow + nanosilica, respectively, the self-Adhesive flowable resin composites used in this research were Fusio Liquid Dentin Self-adhesive flowable composite (Pentron Clinical, Orange, CA, USA) and Vertise Flow Self Adhering Flowable Composite (Kerr Corporation), serving as control groups. The makers of Vertise Flow assert that their product uses adhesive technology from OptiBond to achieve two distinct but complementary binding strengths with the tooth structure: first, via the chemical interaction between the tooth's calcium ions and the phosphate functional groups of a GPDM monomer, and second, through mechanical anchoring. Further, the dentin smear layer and the interpenetrating network generated by the polymerized monomers of Vertise Flow and collagen fibers create a micromechanical connection. The principal chemical bonding process, as stated by Kerr, is the interaction of tooth-derived calcium ions with phosphate functional groups in the GPDM monomers. You might recognize this chemical from





OptiBond. As with many self-etching materials, the etching process is aided by a pH of 1.9, indicating a micromechanical secondary bonding mechanism. However, it is said that following light curing, this pH would rise to almost neutral. Fusio is a flowable composite containing glass fillers and nano-sized amorphous silica, according to the manufacturer. 4-META stands for 4-methacryloxyethyl trimellitic acid. The distinctive acidic (low pH) and hydrophilic formula of Fusio Liquid Dentin sets it apart from the competition. When the methacrylate monomers come into contact with the tooth's surface, the negatively charged carboxylic acid groups hold the mineral ions in the tooth structure together. Polymerization of monomers into dentin improves dentin bonding and sealing capacity by neutralizing carboxylic acid groups.

There has been encouraging progress in adhesive dentistry with the advent of self-etch adhesives. These adhesives have the potential to chemically interact with collagen fibers covered with hydroxyapatite and to reduce the number of application steps required. Reduced or eliminated postoperative discomfort and reduced moisture sensitivity are two additional benefits of these products [20]. It's possible that acid-eroded tooth surfaces might benefit from coatings made of these self-Adhesive flowable resin composites [21]. The presence of oxygen can reduce their polymerization. Some elements seem to be associated with this adhesive's high performance. The hydroxyapatite crystals can be preserved, and the smear plugs can be dissolved at a lower pH, and the monomers can more easily penetrate and polymerize in the underlying dentin to create a sufficient hybrid layer. In addition, the acidic primer and adhesive also include photoinitiators, which enhance the efficiency of monomer polymerization and make solvent evaporation easier [22]. Some have hypothesized that the chemical interaction between dimethacrylate Phosphate Monomer (MDP) and the tooth tissues, together with its high hydrolytic stability, could help the bond strength last for a long time [23]. Also, when the microbrush is mechanically compressed during agitation, it has a better chance of dissolving the smear layer. The molecules are also more vigorously stirred, which increases the solvent's evaporation and the monomer's diffusion into dentin. This, in turn, increases the percentage of polymer formation and improves the adhesive layer's mechanical properties.

Improving bond maturation necessitates first determining the short-term bonding effectiveness of adhesives (after 24 hours), which represents the initial setting period and served as the baseline data for comparison with the other aging periods. The second stage of aging occurred three months later. For three months, the specimens were kept at room temperature in distilled water, with the water being replaced every three days. Some articles found that after a year of water storage, the resinous components in the hybrid layer are extracted, leading to a considerable decrease in bond strength [10, 11]. However, the influence of storage conditions and duration on adhesive bond strength varies depending on the material [24].

Two distinct processes account for water's detrimental effect on nanocomposites. The first process is the enlargement of the nanocomposite's matrix volume, which occurs when water molecules transform the material's behavior from an elastic to a plastic state. The material's stiffness is reduced following this mechanism. The second process involves the breakdown of nanocomposite materials when exposed to water. Research has demonstrated that the mechanical characteristics, such as elasticity modulus, of restorative composites and nanocomposites may be affected by humidity in a variety of ways, with the effects being temperature- and time-dependent. Prior research has shown mixed results when it comes to the effects of soaking dental composite samples in water for different amounts of time and





temperatures. Some studies found an increase in elasticity modulus [25, 26], while others found a reduction [27]. It has been shown that keeping dental composites—in humid environments does not affect their elasticity modulus in some circumstances [28, 29]. Nevertheless, the findings from this study and the ones from prior research [25, 29] showed that water's effect on dental nanocomposite mechanical characteristics is type- and storage-circumstance-dependent and does not follow a predefined law. One potential side effect of nanocomposite materials is increased water absorption and the subsequent deterioration of the resin matrix interface, which is a consequence of the nanoparticles' high surface-to-volume ratio [29]. With a p \leq 0.001, the following order of statistical significance was seen after 24 hours: Fusio Control (17.31±2.66 MPa), Fusio+Nanosilica (19.08±1.45 MPa), Vertise Control (21.52±1.15 MPa), and finally, Vertise+Nanosilica (23.53±1.56 MPa). After three months, there was no significant difference between the Vertise control and Vertise + Nanosilica groups, with the lowest statistical mean μ -Shear Bond Strength (MPa) as recorded by Fusio Control (12.83±1.88 MPa), followed by Fusio + Nanosilica (16.99±2.01 MPa), and finally Vertise Control (18.8±1.47 MPa).

According to the earlier findings, the changed samples had a greater u-shear bond Strength (MPa) compared to the unmodified samples of the identical material. This demonstrates that increasing the flexural modulus of elasticity is the most noticeable advantage of using nanoparticles. While most samples show greater average bond strength values when compared to the control, unaltered sample, this difference is not statistically significant. The findings of the shear bond tests demonstrated that the bond strengths of the self-adhesive flowable composite were comparable to those of dentin and enamel, and that the bond strengths were further enhanced after selective acid etching [30]. Because GPDM contains both a hydrophilic acidic phosphate group and a short spacer group, changes in the volume or dimensions of the flowable composite resin can have major impact on the bonding performance of the material. Among other self-etch adhesive solutions, Bektas et al. [7] reported that Vertise's SBS to dentin was tested. Although the researchers found that OptiBond All-In-One's shear bond strength was 27 MPa and Vertise's was 25.1 MPa, the variation was not substantial. Varying fillers might weaken Vertise's binding strength. Due to the increased viscosity of filled resins, Miyazaki et al. [31] hypothesized that the adhesive resin's filler could lessen the dentin surface's wetting. This would weaken the shear bond strength by lowering the monomer penetration. An important factor affecting the mechanical characteristics of nanocomposites is their microstructure. The behavior of the composite is influenced by the volume percentage. the aspect ratio, and the interaction of the nanoparticles with the matrix. Interactions with nanoparticles have the potential to change the molecular structure of the resin matrix at the contact [32]. Nanoparticles, according to Beun and coauthors [33], may improve the flow and mechanical characteristics of flowable composites. According to earlier research, the physical characteristics of nanocomposites may be improved, and the shrinkage caused by polymerization can be significantly reduced by increasing the proportion of nanofiller [34]. The mechanical property results show that agglomeration, in which several nanosilica particles are bound together, can provide a non-uniform distribution of nanoparticles, which can cause non-homogenous strengthening [17]. The presence of agglomerates at greater nanosilica concentrations has been indirectly confirmed.

5. CONCLUSIONS

The addition of the Nanosilica particles to the self-adhesive materials used in this study might have a positive effect on the shear bond strength.





6. RECOMMENDATIONS

Further studies are required, however, regarding the hazards of nanosilica versus the benefits of larger sample sizes, the delivery mechanism and mode of action.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

FUNDING

No funding

AUTHOR CONTRIBUTIONS

Conceptualization, MFH, MMZ, and HEET, Methodology: HAAE and HOF, investigation and formal analysis, visualization, validation, and writing original draft preparation: MFH, HEET and MMZ, Reviewing and editing: MMZ. All authors reviewed the manuscript.

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APPENDIX: LIST OF ABBREVIATIONS

4 META 4-methacryloyloxyethyl trimellitate anhydride

DEJ Dentino-enamel junction
DENJ Dental enamel-dental junction
GPDM Glycerophosphate dimethacrylate
HEMA 2-Hydroxyethyl methacrylate.
HEMA Hydroxy-ethyl methacrylate

MDP 10-methacryloyloxydecyl dihydrogen phosphate

MPa Mega Pascal SD Standard deviation

TEGDMA Triethyleneglycol dimethacrylate
TEOS Transesterified orthosilicate
UDMA Urethane dimethacrylate.