Enhancing flowability and wettability of dental composites using copper nanoparticles

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Dental composites are popular in restorative dentistry due to their aesthetics, mechanical strength, and versatility. However, optimizing viscosity, flowability, and wettability in their application is challenging. Copper nanoparticles have garnered significant attention in dentistry due to their unique properties, including antimicrobial and anti-cariogenic effects. This study investigates the incorporation of copper nanoparticles into dental composites, focusing on their impact on the dental composite properties and its potential clinical applications. Characterization methods were utilized to provide comprehensive data about the physical and structural properties of the modified dental composites. Adding copper nanoparticles reduced the dental composite viscosity, resulting in improved flowability and wettability measured by the contact angle. The dental composites modified by copper nanoparticles showed more hydrophilic properties. These enhancements make the modified composites particularly suitable for use as a liner in deep cavities or as a luting agent or root canal sealer during root treatment or orthodontic appliance adhesion. As CuNP concentration increases, the contact angle decreases, indicating reduced hydrophobicity and enhanced wettability. Observations of polymerization inhibition suggest a viscosity decrease, transitioning the composite from a semisolid to a more flowable state. The used statistical model confirms a consistent decline in contact angle with each 1% increase in CuNP concentration. This tunable surface behavior suggests that higher concentrations of CuNPs enhance hydrophilicity, potentially improving dental composite spreading and dental bond adhesion. These findings highlight the versatility of CuNP-modified composites for targeted dental applications.

Keywords: Copper nanoparticles; dental composite; flowability; contact angle; viscosity

INTRODUCTION

Dental composites are widely used in restorative dentistry due to their aesthetic appeal, mechanical strength, and versatility. However, achieving optimal viscosity, flowability, and wettability remains a challenge in their application [1]. These properties ensure proper adaptation to cavity walls, ease of handling, and enhanced clinical outcomes. Recent advancements in nanotechnology have introduced innovative methods for modifying material properties by incorporating nanoparticles. Copper nanoparticles (CuNPs) have gained attention in polymer science due to their unique physical, chemical, and antimicrobial properties [2, 3]. Significant attention is paid to polymer science due to its unique physical and chemical properties. These nanoscale particles, known for their high surface area and catalytic activity, play a crucial role in altering the rheological behavior of polymer matrices, allowing them to interact effectively with dental composites [4]. The key effect of incorporating CuNPs into polymers is a noticeable reduction in viscosity. This behavior arises from the nanoparticles' ability to interact with polymer chains, disrupting their entanglements and enhancing flow characteristics [5, 6]. Such a reduction in viscosity simplifies polymer processing methods like extrusion, injection molding, and coating, making them more efficient, especially for those polymers used in dental fields and clinical applications such as e; viscosity dental composites, which need to have good surface wettability properties to be sure that the restorations

have no marginal leakage which could enter bacteria

and further secondary caries or infection occur.

Additionally, the decrease in viscosity contributes to improved dispersion of nanoparticles in polymer nanocomposites, ensuring uniform properties and enhanced material performance. With their thermal conductivity [7], antimicrobial properties, and capacity to modify flow behavior, CuNPs are valuable additives for developing advanced, multifunctional polymer systems. Furthermore, the anti-carogenic properties of CuNPs add a therapeutic dimension to the material to be used inside the root canal and as a liner in deep dental cavities [8]. This study explores the incorporation of copper nanoparticles into dental composites and shows their effect on their viscosity, flowability, and wettability. Also, we examined the mechanisms through which CuNPs influence the dental composite polymer viscosity, emphasizing their advantages and potential applications across various industries, including manufacturing and biomedicine [3, 9]. The findings provide a novel approach for improving the performance of dental composites, addressing both clinical and material properties perspectives to facilitate the manipulation of dental composites and using them as luting agents or root canal sealers[10]. Developing advanced dental composites has been a cornerstone of restorative

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dentistry, providing aesthetic and functional solutions for dental restorations. Among the key challenges in optimizing these materials are achieving desirable flowability and wettability, critical for precise application, adaptation to cavity walls, and minimization of voids. Enhancing these properties facilitates clinical manipulation and improves the restorations' overall durability and longevity [11]. Copper nanoparticles (CuNPs) have emerged as a promising additive in dental composites owing to their unique physical, chemical, and biological properties. Known for their high surface area, catalytic activity, and antimicrobial characteristics, CuNPs offer significant potential for modifying the rheological behavior of polymer matrices. When incorporated into dental composites, CuNPs interact with polymer chains, disrupting entanglements and reducing viscosity, improving flowability and wettability [12]. This enhanced material performance ensures better adaptation to intricate cavity geometries, making CuNP-modified composites ideal for applications such as luting agents, liners in deep cavities, and root canal sealers.

Additionally, the antimicrobial properties of CuNPs add a therapeutic dimension to the material, contributing to secondary caries prevention and improved oral health outcomes [13, 14]. This study explores how CuNPs influence dental composites' viscosity and flow characteristics. By addressing both clinical and material science perspectives, it aims to present a novel approach to advancing the functionality and versatility of dental restorative materials. The findings could pave the way for the development of multifunctional dental composites with enhanced handling properties and therapeutic benefits.

MATERIALS AND METHODS Materials

- Light cure meta dental composite with organic matrices Bis-GMA, Bis-EMA, Urethane methacrylate, and trimethylene glycol methacrylate. Range of dimensions of Inorganic silica filler particles 0.01 to 0.7 µm. Percentage by volume of total inorganic Silica fillers wt. 75%.
- Halogen/LED light intensity of more than 400mV/cm² and wavelength of 430 ~ 480 nm.
- Clean and dry glass slides.
- Fifty grams of metal weight for initiation of the flow.

- Whatman Grade 4 Qualitative Filter Papers (20-25 μm particle retention).
- Ultrasonic tips for oscillation and mixing.

Copper Nanoparticles Synthesis

Copper (II) nanoparticles were synthesized using the Turkevich method (1950), and seven CuNP concentrations were prepared. Then, 0.1 gm of the silica-reinforced dental composite was prepared. A control group was established using pure composite. The CuNPs were synthesized by the Turkevich method using copper (II) sulfate pentahydrate as a precursor salt and starch capping agent [15]. 0.1 M copper sulfate pentahydrate solution was added to 120 mL starch solution (1.2%) with continued stirring for 30 minutes, and 50 mL of 0.2 M ascorbic acid solution was added to the synthesis solution under continuous fast stirring. 30 mL of 1 M sodium hydroxide solution was slowly added to the solution while stirring at 80 °C for 2h on a hot plate. When the solution turned yellow after the reaction, the solution was allowed to stay for 12 hours at room temperature. The precipitates were extracted from the solution by filter paper and washed with distilled water and 70% ethanol 4 times to remove the excessive starch on the nanoparticles. The Yellow ocher color precipitates were left to dry at room temperature. The prepared nanoparticles were stored in a glass vial for the experiment [16].

Dopping and oscillation of the copper nanoparticles in the composite matrix

The air-dried (CuNPs) were mixed into the composite using a plastic spatula on a glass slide. Then, oscillation with an ultrasonic tip forms a homogeneous mix with different concentrations. Six CuNP concentrations were prepared: 10 %,20%, 30%,40%, 50%, and 60%. For each sample, 0.1 gm Meta dental composite. A sample of the nonmodified composite was used as a control. Each sample was placed between 2 glass slides, and a standard 50 gms compressive load was applied, allowing the composite to flow and wet the glass surface. The contact angle was measured for each specimen as an indirect measure of viscosity change [17]. Contact angles were measured, analyzed, and compared to the control, and each sample was on a standard baseline during contact angle measurements [18].

Characterization Techniques

The characterization and testing of the copper nanoparticle-modified dental composites were

conducted using multiple analytical techniques and instruments. Raman spectroscopy was performed using a Renishaw inVia Raman Microscope (Renishaw plc, UK) with a 785 nm laser source. The spectra were collected in the 50-3500 cm⁻¹ range with a resolution of 1 cm⁻¹, operating at room temperature. This analysis was crucial for understanding the molecular interactions between the copper nanoparticles and the composite matrix and confirming the successful incorporation of CuNPs into the dental composite.

X-ray diffraction (XRD) analysis was carried out using a Bruker D8 Advance diffractometer (Bruker AXS, Germany) with Cu K α radiation (λ = 1.5406 Å). The diffraction patterns were recorded in the 20 range of 10-80° with a step size of 0.02° and a scanning rate of 2° per minute. The XRD analysis was essential for determining the crystalline structure of the silica components and the incorporated copper nanoparticles, providing information about phase composition and crystallite size. Contact angle measurements were conducted using a Drop Shape Analyzer (Krüss GmbH, Germany) at room temperature (23 \pm 1°C) and controlled humidity (50 \pm 5%). For each concentration of CuNPs (0%, 10%, 20%, 30%, 40%, 50%, and 60%), the composite samples were placed between two glass slides under a standardized 50-gram load. The contact angle was measured using the sessile drop method, with three replications per treatment, to ensure statistical validity. Images were captured using a high-resolution camera and analyzed using the device's proprietary software to determine the contact angle values.

These characterization methods were selected to provide comprehensive data about the modified dental composites' physical and structural properties. The Raman spectroscopy offered insights into molecular interactions and chemical bonding, while XRD analysis confirmed the crystalline structure and phase composition. Contact angle measurements provided quantitative data about the composites' wettability and flow characteristics, crucial parameters for clinical applications such as cavity linings and root canal sealing.

Statistical Analysis

The contact angle data were presented as Means of 3 replications per treatment. The correlation coefficient was calculated. Analysis of variance (ANOVA) was used to test the significant differences at $P \le 0.05$. A post-hoc test was done. Linear regression analysis was done using the statistical software Minitab Inc. (2007).

RESULTS AND DISSCUSSION Charecterization Techniques

First Raman Spectrum (Image 1): Silica (SiO₂) only: Raman Spectra and Raman confocal images conform to formed silica without any additives. Figure 1 shows the silica skeletal mode backbone's characteristic Raman shift band formation. The key peaks are 50-150 cm⁻¹. These low-frequency peaks correspond to lattice vibrations often seen in amorphous or crystalline materials like silica. The 608, 637 cm⁻¹ in the Raman spectrum region is characteristic of silica due to Si-O-Si bending modes. The 1111 cm⁻¹ is a strong peak typical for the asymmetric stretching mode of Si-O-Si bonds in silica. The 1451, 1602 cm⁻¹ peaks appear due to possible surface impurities or carbon-based contamination, not intrinsic to pure silica. The 2871, 3089 cm⁻¹ is a high-wavenumber range related to C-H stretching, which could be due to adsorbed organic species or contamination. The first spectrum predominantly represents silica (SiO₂), likely in an amorphous or mesoporous form [19].

Second Raman Spectrum (Image 2): Silica with Cu Nanoparticles: The second spectrum corresponds to silica (SiO₂) with Cu nanoparticles, likely a composite material where the copper enhances Raman scattering signals from the silica framework [20]. Figure 2 shows due to phonon vibrations, copper nanoparticles show peaks in the low Raman shift region (<200 cm⁻¹). Their presence may also enhance nearby Raman peaks (SERS effect), amplifying silicarelated modes. The 141.42 cm⁻¹ peak is significant and aligns with the phonon modes of Cu nanoparticles, indicating their presence. The 1293, 1602 cm⁻¹ additional peaks indicate possible defects or surfaceenhanced Raman scattering (SERS) effects caused by the Cu nanoparticles interacting with silica [21]. The 1711.32 cm⁻¹ peak could reflect further Cu vibrational modes influenced by local interactions. The 2871-3089 cm⁻¹: C-H stretching, potentially from organic residues or functional groups on the silica-Cu composite surface. Finally, the 608, 637 cm⁻¹, and 1111 cm⁻¹ peaks are consistent with the bending and asymmetric stretching mode of Si-O-Si bonds in silica (as spectrum 1). The surface topography and morphological characteristics of pure silica and copper nanoparticle-incorporated silica were investigated using 3D Raman mapping. The analysis revealed distinct differences in surface features and structural organization between the two samples. For pure silica in Figure 3, the 3D topographical mapping showed a relatively uniform surface with moderate roughness, displaying peak heights up to 11094.3 µm along the Z-axis.





Figure 2. Second Raman spectrum peaks and image of silica and copper nanoparticles.



Figure 3. Three-dimensional Raman confocal mapping of pure silica composite surface.



Figure 4. Three-dimensional Raman confocal mapping of copper nanoparticle-modified silica composite surface.

The surface exhibited a granular texture with evenly distributed particulate features, indicating the homogeneous nature of the silica matrix. The spatial distribution of these features across the X-Y plane (951.3 μ m × 704.8 μ m) demonstrated consistent particle size distribution and surface regularity, characteristic of well-dispersed silica particles in the composite matrix.

In contrast, the copper nanoparticle-incorporated silica in Figure 4 displayed significantly altered surface characteristics. The Z-axis measurements revealed increased surface heights up to 28842.7 μ m, indicating a substantial modification in surface topology following CuNP incorporation. The surface exhibited more pronounced vertical features with sharper peaks and deeper valleys, suggesting the formation of complex hierarchical structures. This

The comparative analysis of both surfaces provides valuable insights into the structural modifications induced by CuNP incorporation; The nearly threefold increase in surface height (from 11094.3 µm to 28842.7 µm) indicates significant vertical growth and structural reorganization. The more pronounced surface features in the CuNP-incorporated sample suggest enhanced surface area and potentially improved interaction capabilities with the surrounding environment. The maintained X-Y dimensions (951.3 μ m × 704.8 μ m) in both samples ensure comparable analysis conditions while highlighting the vertical structural modifications. These surface characteristics directly correlate with the observed changes in flowability and wettability, as the increased surface complexity and roughness contribute to the modified physical properties of the dental composite. The enhanced surface area and structural organization may also play a crucial role in the material's improved performance as a dental composite, particularly in terms of adhesion and integration with tooth structures.

XRD Characterization

The XRD of pure silica is depicted in Figure 5. The spectra showed that the broad X-ray diffraction pattern shows a Low-Cristobalite (SiO₂) crystal structure. A series of 20 angles, d-spacing values, and relative intensities of diffraction peaks are provided. The most intense peak occurs at 22.019° (d = 4.033 Å) with 100% relative intensity, and the reflection from prominent planes include (-1,0,-1), (-1,-1,-1), and (-2,0,0). The SiO₂ structure having primitive lattice with lattice parameters (a = 4.964 Å, c = 6.920 Å and, c/a Ratio = 1.394). Figure 6 shows the X-ray diffraction pattern silica with copper nanoparticles. The most intense and characteristic for copper cubic crystal structure peak was observed at $2\theta = 43.718^{\circ}$ (d = 2.0689 Å), intensity = 100%, and the reflection from (-1,-1,-1) plane. Other notable peaks at $2\theta = 50.926^{\circ}$ (d = 1.7917 Å), 74.893° (d = 1.2669 Å). The pattern shows the low quartz (SiO₂) with the hexagonal crystal structure. The most intense peak at $2\theta = 26.644^{\circ}$ (d = 3.3430 Å) Intensity = 100% and reflection from (-1,0,-1) plane . Additional peaks at $2\theta = 20.861^{\circ}$ (d = 4.2548 Å), 36.550° (d = 2.4565 Å). The SiO₂ structure has a primitive lattice with lattice parameters a = 4.9130 Å and c = 5.4040 Å



Figure 6. The X- ray diffraction of copper and silica

Effect of copper nanoparticles on wettability

The present study tried to highlight the concept of altering the physical properties of the current dental composites by implementing nanoparticles. Changing the physical properties as viscousity can play a critical role in raising the success of dental endodontic activities. The silica-based dental composite was selected for its mechanical and optical properties. This polymer was suitable for handling easily and was compatible with CuNP treatments. Mixing these tiny nanoparticles with the composite at an increasing rate from 10 to 60% was negatively correlated to its viscosity (r=-0.97) Figure 7 as there is a direct relationship between viscosity and the contact angle of the matter[22].

The composite's viscosity was indirectly determined by recording the composite borders' contact angle on a glass slide. The contact angle of the composite was measured as 160.96° before adding CuNPs, indicating that the surface is highly hydrophobic, as shown in Figure 8A. A significant decrease in the contact angle of the composite was recorded in Figure 8B upon adding CuNPs at a rate of 10%. Further increase of the CuNP resulted in a drastic decline in the measured contact angle, revealing an increase in flowability of the used dental composite, as depicted in Figure 8C. A further increase in CuNP concentration of up to 30% insignificantly changed the contact angle, as shown in Figure 8D. Another drastic drop in contact angle measure was recorded upon the increase of CuNPs



Figure 7. Effect of the CuNPs concentration on the contact angle measurements of the treated dental composites. Data presented as means, Bars refer to S.E. Significantly different means were denoted by different alphabetical letters, $p \le 0.05$.



Figure 8. Contact angles at diffrent concentrations of CuNPs in dental Composite (A= control 0%CuNPs, B= 10%, C=20%, D=30%, E=40%, F=50% and G= 60%).

concentration to 40 & 50 %, releasing a more wettable composite with insignificant effect between the two treatments represented in Figure 8E& F. Surprisingly, raising the CuNP concentration to 60% significantly reduced the contact angle with a loss of symmetry in the imaged drop. Both sides recorded a low but different contact angle measure, proposing a different distribution of the composite material and CuNPs recorded in Figure 8G. Resin-based endodontic sealers are critical in achieving a hermetic seal within the root canal system. Their importance lies in their ability to flow into intricate anatomical irregularities, accessory canals, and dentinal tubules, often inaccessible during mechanical instrumentation. The flowability of resin-based sealers ensures that gaps between the gutta-percha core and the canal walls are minimized, reducing the risk of microleakage and bacterial infiltration. This property is crucial for maintaining long-term success in root canal therapy by preventing reinfection. Additionally, their excellent adhesion to dentin enhances the bond strength and stability of the obturation. Resin-based sealers are also radiopaque, allowing for post-operative evaluation. Their ability to set within the canal without excessive shrinkage further supports the integrity of the seal. Proper flowability and biocompatibility make resin-based endodontic sealers a reliable choice for achieving optimal endodontic outcomes[23].

Changing physical properties such as viscosity can play a critical role in increasing the success of dental activities. Before curing, endodontic dental composites behave as viscous semisolids, an intermediate phase with dual flowability and wettability properties on surfaces like enamel and dentine or even glass and ceramics [24]. Copper's inhibition of polymerization is primarily based on its ability to interfere with free radical chain reactions, a mechanism relevant to processes such as autoxidation or polymerization in the presence of radicals [25]. The following is the effect of polymerization inhibition by copper nanoparticles:

 Radical Scavenging: Cu(II) and Cu(I) can interact with free radicals generated during polymerization. These ions act as catalysts in redox reactions, which lead to the termination of radical species and halt the propagation of polymer chains. Cu(II) reacts with active radicals (e.g., R• or ROO•), converting them into inactive species. Reaction Cu(I), in turn, can be oxidized back to Cu(II) while interacting with peroxides (ROOH), decomposing them into non-radical products [26].

- Peroxide Decomposition: Copper ions catalyze the breakdown of hydroperoxides (ROOH), precursors of free radicals. This reduces the formation of new radicals that propagate the polymerization reaction [27].
- Redox Cycling: Copper's ability to switch between Cu(I) and Cu(II) states allows it to continuously deactivate radicals, making it a potent inhibitor in radical-driven processes like polymerization [28].
- Chelation Effects: Copper ions often form complexes with oxygen-containing species or other ligands, stabilizing reactive intermediates and preventing chain initiation or propagation [29].

Clinical applications of CuNPs modified dental composites

The tunable flowability of CuNP-modified dental composites offers several promising clinical applications: (i) Improved dental restoration adhesion, high CuNP concentrations (40-60%) enhance hydrophilicity, which may improve the bonding efficiency of adhesives and composites to dentin and enamel, leading to stronger and more durable restorations. (ii) Enhanced dental composite spreading, increased wettability can aid in the even distribution of dental composites, reducing voids and improving marginal adaptation in restorative procedures. (iii) Antibacterial and bioactive potential, CuNPs are known for their antimicrobial properties. Incorporating them into dental composites may help reduce bacterial colonization, lowering the risk of secondary caries and infections. (iv) Hydrophobic coatings as protective barriers, low CuNP concentrations (0–10%) can be used in hydrophobic coatings for sealants and protective layers, enhancing resistance to moisture and biofilm formation. (v) Periodontal and endodontic applications, the modified surface properties could be beneficial in periodontal treatments, such as guided tissue regeneration membranes, and in endodontic sealers, where controlled wettability influences material performance. (vi) Potential role in prosthodontics and orthodontics, CuNP-modified materials may improve the adhesion of prosthetic devices and orthodontic brackets, leading to better retention and reduced failure rates. (vii) Extended shelf life of polymeric materials used in dentistry, copper ions prevent the oxidative degradation of polymers like polyethylene by terminating free radical chains. Copper stabilizes monomers (e.g., styrene or acrylates) during storage to avoid unwanted premature polymerization [30, 31].

Finally, this novel approach demonstrates the promising potential for improving dental composites' functional and therapeutic performance and manipulation in restorative dentistry by confirming the significant negative correlation between CuNP concentration and the contact angle of the prepared dental composite, thereby rejecting the null hypothesis.

CONCLUSION

There is a significant and negative correlation between the CuNP concentration and the contact angle of the prepared dental composite thus rejecting the null hypothesis. As the percentage of copper nanoparticles increases, the contact angle decreases. The apparent observations of polymerization inhibition indicated the reduced viscosity as the dental composite transformed from semisolid to flowy upon increasing the CuNP concentration. The mean contact angle indicates a general trend toward reduced hydrophobicity and increased wettability of the modified composite. There is wide variability in surface behaviour due to Nano Cu addition. The linear regression model quantifies the relationship, showing a consistent decrease in contact angle for every 1% increase in CuNP concentration. Low concentrations of CuNPs (0–10%) can be applied for hydrophobic coatings. High concentration of CuNPs (40-60%) may be suitable for hydrophilic applications like water spreading and Adhesion. This study demonstrates the tunability of surface properties using Nano Cu, enabling targeted applications across dentistry. Building on these findings, future research work can explore the long-term stability and mechanical properties of CuNP-modified dental composites to assess their clinical use. Investigating the biocompatibility potential and anti-cariogenic benefits of CuNPs at varying concentrations could enhance their applicability in dentistry.Additionally, optimizing CuNP dispersion techniques may improve uniformity and consistency in surface behavior. Exploring hybrid nanoparticle formulations by combining CuNPs with other bioactive materials could provide enhanced performance for specific dental applications. Beyond the dentistry field, the tunability of surface properties demonstrated in this study suggests potential applications in biomedical coatings, antifouling surfaces, and regenerative materials. Future studies should also examine the effects of different polymer matrices on the wettability and functional properties of CuNP-integrated composites. By refining formulation strategies and conducting in vivo studies, the practical implementation of CuNP-based dental materials can be further advanced, showing the way for improved adhesion, antimicrobial protection, and enhanced clinical outcomes.

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