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The Impact of Silanized Graphene Nanoparticles on Selected Mechanical and Physical Properties of a Heat-Cured Acrylic-Based Soft Liner

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ABSTRACT

Background and Objective: Soft liners are polymers that provide comfort to the tissues by acting as a cushion between the soft tissue surface and the hard acrylic. Since soft liners have demonstrated numerous drawbacks, the properties of soft lining materials must be enhanced. This study aimed to assess the effects of different proportions of silanized graphene nanoparticles on the mechanical and physical properties of soft denture lining materials, including shear bond strength, thermal conductivity, and water sorption and solubility.

Methods: In this laboratory study, 90 samples were fabricated and divided into three groups, each comprising three subgroups (n=10). The control groups consisted of soft liners made of heat-cured acrylic without silanized graphene nanoparticles, while the experimental groups consisted of soft liners made of heat-cured acrylic containing 0.5% and 1% silanized graphene nanoparticles. Disc-shaped soft liner samples with a diameter of 50 mm and a thickness of 0.5 mm were produced and evaluated according to ISO 20795-1:2013 (E) for the physical assessment of solubility and water sorption.

Findings: Shear bond strength was recorded as 0.54±0.39 in the control group compared with 0.54±0.29 in the 0.5% group (p=0.000). The thermal conductivity test demonstrated a significant increase in mean values, from 0.18±0.01 in the control group to 0.55±0.04 in the 1% group (p=0.000). In contrast, the water sorption and solubility test revealed a decrease in mean values.

Conclusion: The results of the present study demonstrated that the incorporation of SGNPs into the heat-cured soft denture lining material was effective in reducing both solubility and sorption levels. Additionally, the mechanical property of shear bond strength was improved. Moreover, the thermal conductivity of the heat-cured soft denture lining material increased significantly as the concentration of SGNPs increased.

Keywords: *Soft Liner, Denture Base, Graphene, Nanoparticles, Nanotechnology*.

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Introduction

Over time, the comfort, effectiveness, retention, stability, and esthetic appearance of dentures may deteriorate, and patients' satisfaction with their existing dentures may decline. Owing to the characteristics of the supporting (foundation) tissues, dentures occasionally require adjustment to achieve a proper fit (1). The ability to distribute loads across the mucosal tissues constitutes the primary functional characteristic of elastomeric soft lining materials. As a result, mucosal injuries are reduced, and masticatory efficiency is improved. This procedure involves modifying and adapting the denture foundation to accommodate gradual changes in the oral tissues, thereby ensuring a proper fit and maximizing patient satisfaction (2, 3). The principal drawbacks of soft tissue liners include their poor thermomechanical properties, a gradual increase in hardness and surface roughness, and inadequate durable bonding to the denture surface (4, 5).

The mechanical and physical properties of various dental materials used in prosthodontics, including PMMA resins and soft denture liners, have been shown to be enhanced by the incorporation of nanoparticles (6, 7). Graphene is a carbon allotrope composed of sp^2 hybridized carbon atoms arranged in densely packed planar sheets that are one atom thick and organized in a honeycomb crystal lattice. This structural arrangement renders graphene the thinnest known material, forming a homogeneous crystal lattice devoid of structural dislocations or voids. Owing to this unique property, graphene exhibits novel physical characteristics (8, 9).

In addition to being an exceptionally lightweight material with a planar density of 0.77 mg/m^2, graphene exhibits numerous exceptional properties, including optical transparency, mechanical strength, electrical conductivity, and thermal conductivity (10). Polymers constitute the most commonly attached molecules to graphene. This widespread application is attributable to their diverse chemical structures, affordability, sustainability, and ease of processing, all of which contribute to improved mechanical strength, electrical conductivity, thermal characteristics, and solubility (11).

The present study was designed to evaluate the effects of varying proportions of silanized graphene nanoparticles on the mechanical and physical properties of soft denture lining materials, including shear bond strength, thermal conductivity, and water sorption and solubility.

Methods

The present laboratory investigation received ethical approval from the Ethics Committee of the College of Dentistry, University of Baghdad, on 22 April 2025 (Project No.: 1042225; Ethics No.: 1042).

Specimen grouping: A total of 90 specimens were fabricated for use in the present study and subsequently divided into three main groups, each of which was further subdivided into three subgroups. The control groups comprised soft liners fabricated from heat-cured acrylic without the addition of silanized graphene nanoparticles, whereas the experimental groups comprised soft liners made from heat-cured acrylic incorporating silanized graphene nanoparticles at concentrations of 0.5% and 1% (n=10 per subgroup). For the mechanical evaluation of shear bond strength, heat-cured acrylic blocks measuring 75 mm× 25 mm × 5 mm in length, width, and depth, respectively, and featuring a stopper approximately 3 mm deep, were utilized to prepare the specimens (12, 13), as illustrated in Figure 1.

Surface Functionalization of Graphene Nanoparticles with a Silane Coupling Agent: One of the most critical yet technically challenging steps in the development of high-quality reinforced materials is the achievement of optimal and homogeneous dispersion of nanoparticles throughout the polymeric matrix. This can be effectively accomplished by functionalizing the graphene (G) surface through silanization with (3-methacryloxypropyl) trimethoxysilane (MPS) (14). In the present study, graphene nanoparticles were

treated with 3-methacryloxypropyltrimethoxysilane (MPS) in order to introduce functional (reactive) groups onto the surfaces of the graphene nanofillers, a process achieved through the chemical reaction between the graphene nanofillers and the silane coupling agent. The graphene powder obtained following the silanization procedure was subsequently characterized using Fourier transform infrared spectroscopy (FTIR) to confirm the surface modification.

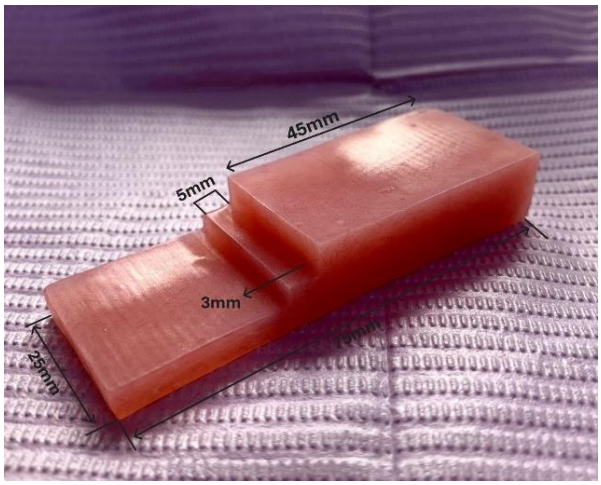


Figure 1. Specimen for shear bond strength test

Nanomaterial: Silanized graphene nanoparticle powder was added to the heat-cured acrylic-based soft liner at two concentrations: 0.5% and 1%. These specific percentages were selected based on the findings of a pilot study that was conducted to determine the most suitable and effective concentration of graphene nanoparticles for incorporation into the soft liner material. According to the technical data sheets provided by the supplier, the graphene nanoparticles have an average particle diameter of 15 microns, a thickness of approximately 8-9 nm, and a specific surface area ranging from 120 to 150 m^2/g.

In accordance with the instrument's specifications, disk-shaped soft liner specimens measuring 40 mm in diameter and 7 mm in thickness were prepared for the physical evaluation of thermal conductivity. Plastic molds were employed during specimen preparation to minimize the likelihood of cracks and other material defects, as illustrated in Figure 2. A total of 10 specimens were prepared for each test subgroup.

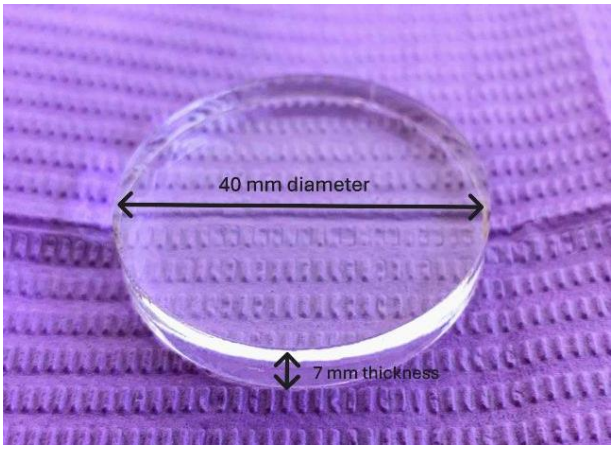


Figure 2. plastic mold for thermal conductivity test

Disc-shaped soft liner specimens with a diameter of 50 mm and a thickness of 0.5 mm were fabricated and subsequently evaluated in accordance with ISO 20795-1:2013 (E) for the physical assessment of solubility and water sorption.

To minimize the risk of crack formation and other structural defects within the material, plastic molds were utilized during the specimen preparation process. For each test subgroup, a total of 10 specimens were prepared, as illustrated in Figure 3.

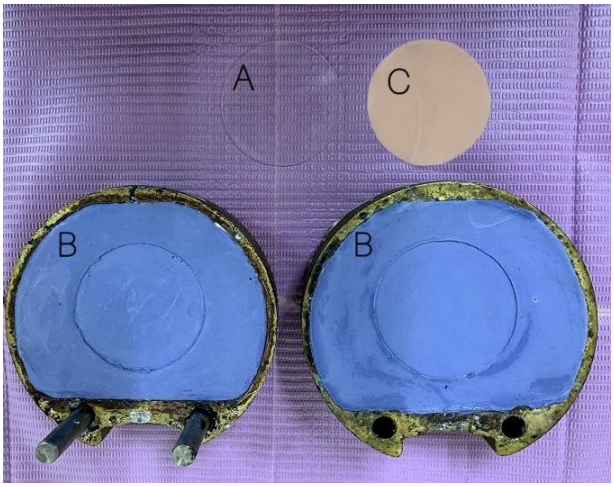


Figure 3. (A) Plastic mold used for the preparation of water sorption and solubility specimens, (B) upper and lower parts of the flask after stone setting, (C) finished test specimens

The thermal conductivity of the specimens was evaluated using a thermal conductivity testing apparatus (Hot Disk). The Instron testing machine was used to test the specimens for shear bond strength, whereas the solubility and sorption of the specimens were calculated as recommended by ISO Standard 20795-1:2013 (E). Silanized graphene nanoparticles were added by weight (0.5% and 1%) to 7.8 g of the monomer of the denture lining material to prepare the experimental group of specimens. The mixture was placed in a clean, dry beaker and mixed using a probe sonication device for three minutes.

The mixture was subjected to vibration at a frequency of 60 kHz and a power of 120 W in order to break up any agglomerates and disperse the nanoparticles into individual particles. To prevent particle aggregation, the soft liner powder was added to the resulting suspension as quickly as possible. In order to maintain a constant powder-to-liquid (P/L) ratio, as specified in the manufacturer's instructions, the weight of the added graphene nanoparticles was deducted from the weight of the soft liner powder.

A statistical advisor was consulted to complete the statistical analysis of the study. The t-test and one-way ANOVA were employed for comparisons between groups, and $p<0.05$ was considered statistically significant.

Results

Shear bond strength test: Shear bond strength values were 0.54±0.39 in the control group and 0.54±0.29 in the 0.5% group (p=0.000, Table 1). The thermal conductivity test revealed a statistically significant increase in mean values, rising from 0.18±0.01 in the control group to 0.55±0.04 in the 1% group (p=0.000, Table 2). Conversely, the water sorption and solubility test demonstrated a decrease in mean values.

Table 1. Comparison of shear bond strength between groups

Groups	N	Mean±SD	F test	p-value
Control	10	0.39±0/54	38.517	0.000
0.5%	10	0.54±0/29	38.517	0.000
1%	10	0.53±0/36	38.517	0.000

Thermal conductivity test: The control group exhibited the lowest mean thermal conductivity value, recorded at 0.1841 W/m·K. In contrast, the specimens containing 1% wt% silanized graphene nanoparticles demonstrated the highest mean value, at 0.5577 W/m·K, followed by the specimens containing 0.5% wt% silanized graphene nanoparticles, which had a mean value of 0.2657 W/m·K.

As shown in Table 2, a statistically significant difference was found among all groups when examined using the one-way ANOVA test.

Table 2. Mean thermal conductivity values and comparison between groups

Group	N	Mean±SD	F test	p-value
Control	10	0.18±0/01	470.164	0.000
0.5%	10	0.26±0/01	470.164	0.000
1%	10	0.55±0/04	470.164	0.000

Water sorption and solubility test: The control group demonstrated the highest mean value, while the lowest mean value was observed in the specimens containing 1% wt% silanized graphene nanoparticles ($p<0.01$), followed by the specimens containing 0.5% wt% silanized graphene nanoparticles (Table 3).

Table 3. Comparison of water sorption between groups

Group	N	Mean±SD	F test	p-value
Control	10	20.29±6/76	5.596	0.011
0.5%	10	15.82±3/03	5.596	0.011
1%	10	13.04±1/56	5.596	0.011

The control group demonstrated the highest mean solubility value, while the lowest mean solubility value was observed in the specimens containing 1% wt% silanized graphene nanoparticles ($p<0.01$), followed by the specimens containing 0.5% wt% silanized graphene nanoparticles (Table 4).

Table 4. Descriptive statistics and one-way ANOVA results for solubility

Group	N	Mean±SD	F test	p-value
Control	10	9.50±4/76	5.481	0.012
0.5%	10	5.82±3/18	5.481	0.012
1%	10	3.89±1/65	5.481	0.012

Scanning electron microscope (SEM): The scanning electron microscopy (SEM) results of the soft liner, obtained before and after the addition of 0.5% and 1% wt% powdered silanized graphene nanoparticles, are illustrated in Figures 4A, 4B, and 4C, respectively.

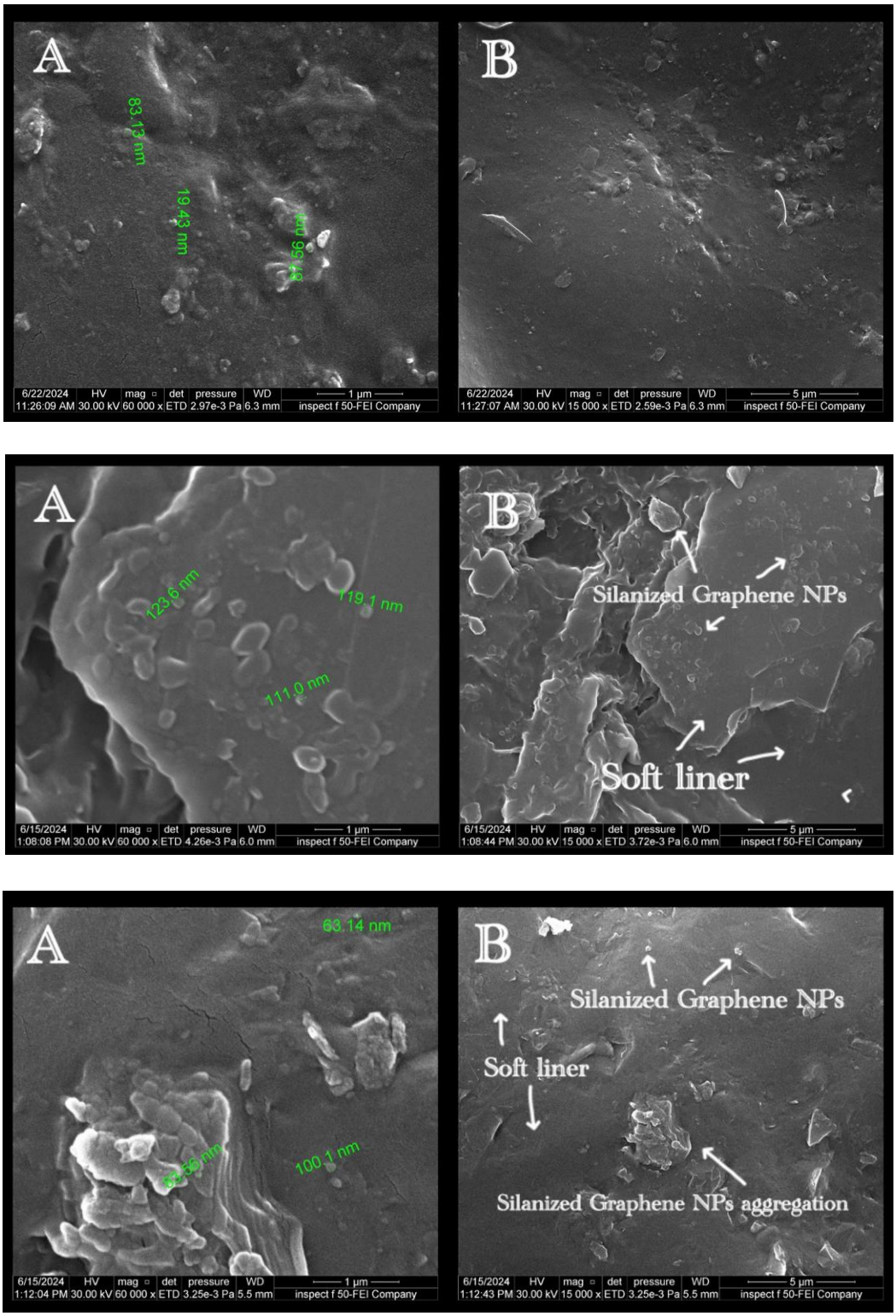


Figure 3. A: SEM images of control soft liner, B: SEM images of the soft liner with 0.5% silanized graphene nanoparticles, C: SEM images of the soft liner with 1% silanized graphene nanoparticles

Discussion

Enhancing shear bond strength was the primary objective of the present study, as this property is of considerable importance in dental materials. Statistical analysis of the test results demonstrated that, compared with the control specimens, the experimental specimens containing 0.5% and 1% SGNPs exhibited a significant increase in shear bond strength.

The most widely used material for denture relining is heat-cured acrylic-based soft denture liner material. Commercially available heat-cured acrylic soft lining materials are generally considered to be far from optimal, owing to their poor tear resistance, low thermal conductivity, and inadequate bonding to the denture base. Consequently, there is a clear need to enhance the properties of these materials (15, 16). Owing to graphene's exceptional mechanical properties, ease of manipulation, functionalization potential, and promising applications in dental and biomedical fields, its use in prosthodontics has been increasingly encouraged (17).

Although denture soft liners are appreciated for enhancing the comfort of removable prostheses, shear stress may occasionally result in debonding between the soft liners and the denture base resin, thereby reducing the lifespan of a relined prosthesis. To improve their performance, two principal strategies are available: surface treatments and the incorporation of materials at the micro- or nanoscale (18, 19).

The increase in shear bond strength following the incorporation of SGNPs may be attributed to the homogeneous dispersion of nanoparticles within the soft liner matrix. By occupying the interstitial spaces between soft liner particles, the nanoparticles function as fillers and thereby increase the surface area available for adhesion to the denture foundation (20). The incorporation of SGNPs into the soft lining material may influence the flowability of the material. The high flowability of the soft lining materials used in this study represents another factor that enhances shear bond strength, as it enables the material to readily adapt to the bonding surface and establish favorable contact (21).

Thermal conductivity refers to a material's ability to transfer heat and constitutes a crucial property of dental prostheses. It is determined by measuring the rate at which heat passes through a material's cross-sectional area (22). According to the results, thermal conductivity increased as the filler ratio increased. This finding may be explained by the observation that, at the 0.5% and 1% ratios, the nanoparticles appeared to overlap one another and form bridging networks, thereby facilitating conductivity (23). Furthermore, the SEM results demonstrated that the elevated thermal conductivity of the specimens could be attributed to the uniform dispersion of SGNPs within the acrylic soft liner, in addition to the intrinsically high thermal conductivity of the nanoparticles themselves.

The results indicate that, within the soft lining material matrix, the nanoparticles are evenly dispersed throughout the specimen, with minimal aggregation (i.e., few clustered groups). Solubility and water sorption were evaluated concurrently by measuring water uptake and the loss of soluble components. The observed decrease in mean water sorption values was consistent with the findings of Al-Hiloh et al. (24), who reported that water sorption decreased with an increasing proportion of AgNPs. Since AgNPs are insoluble in water, they may reduce the total volume of the absorbent polymer. Polymers with higher crosslinking density have been shown to exhibit lower sorption values, as there is less free space within the network structure for water molecules to occupy (25).

The results of the solubility test revealed a highly significant decrease in solubility. As demonstrated by the present study, an increase in SGNP concentration results in a reduction in the percentage of the resin matrix present within the material, which may provide an explanation for the observed decrease in solubility as SGNP concentration increases. This restriction in the amount of absorbed water would be expected to diminish both the potential for molecular adaptability and the leaching of soluble components from the polymer mass (26, 27).

The findings demonstrated that the incorporation of silanized graphene nanoparticles into the heat-cured soft denture lining material successfully reduced both sorption and solubility levels. Additionally, the shear bond strength was improved. Moreover, the thermal conductivity of the heat-cured soft denture lining material increased significantly with increasing SGNP concentration.

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