Research Article

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The Impact of Modified Metallic Nanoparticles on Thermomechanical Properties of PMMA Soft Liner

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Abstract

The acrylic-based heat-cured soft denture lining material is the most commonly used in relining dentures. This material has poor thermomechanical properties which is a disadvantage. This research aimed to study the effect of the addition of modified metallic nanoparticles on glass transition, modulus of elasticity, and coefficient of thermal expansion and contraction of the acrylic soft liner. Alumina nanoparticles were first modified by a silane coupling agent and then added to a soft denture liner powder in different weight percentages (0, 0.5, 1, and 1.5 wt%) using a probe ultrasonication machine for mixing. 120 samples of acrylic-based soft liner were constructed and divided into four groups G1–G4 (n = 30). Each group was in turn subdivided into 3 subgroups (n = 10) according to the test performed. The mean value, SD, Kruskal-Wallis test, and Dunn's Multiple Comparison tests were used to analyze the results statistically. Incorporating 0.5% by weight alumina nano-fillers into acrylic-based heat-cured soft denture lining material, increased the glass transition temperature significantly ($p \leq 0.01$). Additionally, it significantly reduced the coefficient of thermal expansion and contraction, especially at 30 ºC, compared to the control group. The E-modulus was also reduced, especially at 50 ºC, compared to the control group. According to the reported results, the polymer nanocomposites possess distinctive material properties that distinguish them from unmodified acrylic-base soft denture lining materials. Nanocomposites have more thermal and mechanical stability than unmodified acrylic-base soft denture lining material especially when incorporating 0.5 wt% Al_2O_3.

Keywords: Acrylic-based soft denture liners; aluminum oxide nanoparticles; salination; thermal behavior; glass transition

Introduction

Soft denture liners were recently introduced in dentistry. It is a polymeric material that underlines a hard denture to absorb the force of mastication and distributes it over a large area of tissues, to obtain a pleasant interface between denture and oral tissues, and to prevent injuries to alveolar ridges from occlusal forces. In addition, it is used after surgery on soft tissue to facilitate its healing under the hard denture [1]. The soft-liner materials are used to reline the fitting surface of the denture with patients who cannot tolerate the hard base of the denture. They can also improve the masticatory function and provide comfort to the patient [2]. However, when used clinically, these lining materials may exhibit physical and mechanical shortcomings such as color alterations, inadequate strength and porosity [3], and retention of tooth Germs due to insufficient cleaning [4]. Additionally, these materials have a low thermal conductivity between the denture base and oral mucosa, affecting the patient's acceptance of the denture. In addition, the denture base's low thermal conductivity mainly affects the health of underlying supporting tissues [5]. Furthermore, it is challenging to maintain these materials' elasticity for an extended period because of their leachable plasticizers [6]. Insufficient bonding strength with underlying acrylic is another drawback related to soft denture liners [7, 8]. For all the above-mentioned limitations, these materials should be changed every 9–18 months rendering them expensive materials [9].

Several studies have been conducted for the enhancement of soft denture liner properties by the addition of different types of fillers. Soft denture liners' physical and mechanical properties are dramatically improved with inorganic nanoparticles, even with minimum amounts [10–12].

Organic and inorganic materials can create strong connections via silane coupling agents. An alkyl chain with a fourth alkyl chain including several organofunctional groups, such as amine, diamine, methacrylate, epoxy, vinyl, chloro, and phenyl, connects the core silicon atom of silane molecules as one to three alkoxy groups. The alkoxy groups can be hydrolyzed into silanol groups by water independently, from moisture in the air, or further adsorbed on the surface of nanomaterials [13].

The large surface areas of the nanoparticles make them an excellent choice when trying to enhance interfaces because of their surface reactivity, surface energy, and chemical reactivity [14]. Aluminum oxide, commonly called alumina (Al_2O_3) is a ceramic material that possesses high thermal conductive properties and strong ionic inter-atomic bonding, giving rise to its desirable material characteristics. It possesses fine dielectric properties, and high hardness, refractoriness, and excellent thermal properties that make it a good choice for a wide range of applications [15, 16]. In dentistry, Alumina is used for the fabrication of dental implants, ceramic abutments, and crowns and bridges [17]. Although it possesses enviable properties, its application as a structural material has been significantly limited by its low-fracture strength and low-fracture toughness [18].

This study aims to investigate the change in glass transition temperature (T_g), elastic modulus, and coefficient of thermal expansion of acrylic soft-liner material after the addition of Al_2O_3 nanoparticles in concentrations (0.5, 1, 1.5)wt% after taking into account the following study hypotheses where

1. The null hypothesis (H_0) asserts that the inclusion of alumina nanoparticles into the acrylic soft-liner material will not affect the mechanical or thermal characteristics.

2. The alternative hypothesis (H_1) postulated that alumina nanoparticles would improve the mechanical and thermal characteristics of the acrylic soft-liner material.

Materials and Methods

To obtain improved interaction between Al_2O_3 nanoparticles with an average particle size of 20–40 nm (NS6130 01-123, Germany) in concentrations (0, 0.5, 1, 1.5)wt% by weight and heat cured polymerized acrylic soft liner matrix (Vertex-soft heat polymerizing, Netherland), the as-received Al_2O_3 were first surface modified with silane coupling agent 3-(methacryloyloxy) propyltrimethoxysilane (MPS) (Tokyo Chemistry Industry Co., Japan) [19], then embedded into heat cured polymerized acrylic soft liner to investigate the change in thermomechanical properties of acrylic soft denture lining material. 120 specimens were fabricated and divided into 4 groups (n = 30) according to the alumina concentration added (G1: 0 wt% Al_2O_3, G2: 0.5 wt% Al_2O_3, G3: 1 wt% Al_2O_3, and G4: 1.5 wt% Al_2O_3). Each group was then further subdivided into three subgroups (n = 10) according to the test performed (glass transition temperature, E-modulus, and coefficient of thermal expansion and contraction). The process steps are presented in Fig. 1.

Modifying the alumina surface

Surface modification of the Al_2O_3 nanoparticles was accomplished using a 0.6% silane coupling agent in acetone [20]. First, 200 g of acetone and 1.2 mL of the coupling agent were separately combined by ultrasonic oscillation (MSK-USP-3N-LD, MTI Corporation, USA) for 10 min. Then, 30 g of Al_2O_3 nanoparticles were added to the liquid mixture and ultrasonically blended for 30 min at ambient temperature. After that, the temperature was raised to

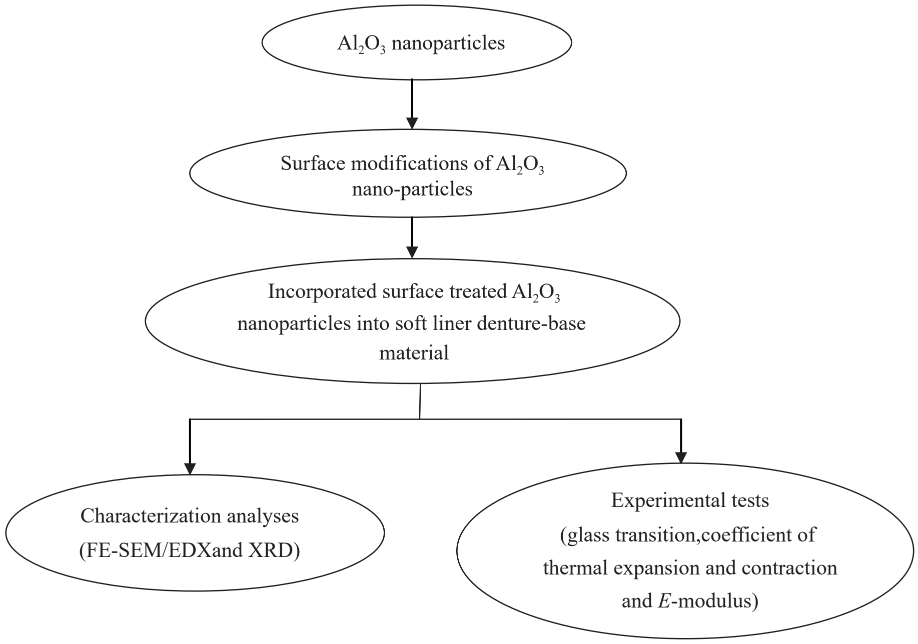

Fig. 1 The process steps.

80 ºC for the coupling process and alternatively dried for 24 h [19].

Al_2O_3 incorporation and mixing

By the manufacturer's recommendations (1.2 g of powder per 1 mL of liquid), the amount of soft denture liner liquid and powder was estimated, and placed separately in a dry, clean glass jar, then the jar was covered with lid. Al_2O_3 nanopowder that was weighed in a clean, dry glass container, and the soft-liner monomer was added to it. This mixture was then combined with a probe sonication instrument to break them into individual nanoparticles by vibration at 120 W and 60 kHz for 3 min [11].

The powder for the soft lining material is then mixed with the monomer containing the nanofillers, packed, and cured as instructed by the manufacturer. To maintain the same manufacturer's P/L ratio, the weight of Al_2O_3 nano-powder should be deducted from the weight of the soft-liner powder [21,22].

All the specimens were completed, polished, submerged in distilled water, and maintained in the incubator at 37 °C for 48 h until testing later after full curing [10].

Characterization analysis

Utilizing X-ray diffraction (XRD) technology (X-rays on, Rayon X, Holand) with CuK_α radiation and a detector operating at 45 kV and 200 mA voltage and current, respectively; scan speed/duration time was 1 deg/min. A fixed time mode of 1.2 s was employed during the data-gathering operation. Scan angles ranged from 2θ = 10°–90°.

Field emission scanning electron microscopy (FE-SEM) equipped with energy dispersive X-ray spectroscopy (EDX) analyses were used to analyze structural and morphological characteristics (Inspect F50, FEI Technologies Inc., USA).

Differential scanning calorimetry

Glass transition temperature (T_g) was measured by the differential scanning calorimeter (DSC) (DSC-60, Shimadzu, Japan). 10 mg powder is obtained by scratching acrylic specimens with a sharp knife were sealed in a standard aluminum crucible in a dynamic air atmosphere (flow rate 25 cm^3/min). The samples were heated from 20 to 190 °C at a rate of 10 °C/min. The chart speed was 20 mm/min [5].

Thermal expansion properties

The coefficient of thermal expansion and contraction was measured by Thermo-Mechanical Analyzer (TMA) (PT1000, Linseis, UN) at 30, 50, and 70 ºC [5].

In the holding chamber, a cylindrical sample of 6 mm in diameter and 15 mm in length, was supported vertically by an optically flat platform. A quartz probe with a constant, minuscule load (about 1 g), guided by a balanced beam, was placed on each sample. To identify the sample's dimensions change, a displacement transducer was attached to the probe. Each sample was weighed, heated at a rate of 10 ºC per minute from 20 to 70 ºC, and then reweighed [23]. The coefficient of thermal expansion and contraction was determined by the following equation [24]:

$$\alpha = \frac{\Delta L}{L_0 * \Delta t}$$

where: α is coefficient of thermal expansion and contraction (ºC^{-1}); ΔL is the change in length per unit length (mm); L_0 is original length (mm); Δt is temperature change (ºC).

Modulus of elasticity

The test was performed as prescribed in the previous test except for the load used. In this test, a load of 50 mg is selected [25]. The computer determines the E-modulus by measuring the ratio of elastic stress to elastic strain [26]:

$$E = \frac{\sigma}{\delta}$$

where E is modulus of elasticity (MPa); σ is stress (MPa); δ is strain.

Statistical analysis

A Kruskal-Wallis test was performed to evaluate the differences between the study groups for each test. Dunn's multiple comparison test was used to compare the mean value of each experimental group. Statistically highly significant (HS) was defined as a probability p value ≤ 0.01. A $p \leq 0.05$ was regarded as significant (S), whilst a $p > 0.05$ was regarded as non-significant (NS).

The statistical package for social sciences, IBM SPSS® software, version 23.0, was used to first computerize and then analyze all of the data.

Results

Field-emission scanning electron microscopy and energy dispersive spectroscopy

The FE-SEM picture of various Al_2O_3 nanoparticles incorporated into the acrylic soft-liner material is shown in Fig. 2. Since no particles were found on the surface of the G0, it seems dense and compact. The surface of the G2–G4 group gets rougher. The white particles that were randomly dispersed on the surface with increasing filler load in G4 might be explained by the presence of numerous agglomerates inside the soft-liner (Fig. 2).

The EDX analysis showed that acrylic-based soft-liner was mainly composed of carbonium (C) and oxygen (O). After the addition of Alumina nanofillers, Aluminum (Al) was detected on the surface of the samples in addition to C and O. The Al and O were increased on the surface of the nanocomposite exponentially with the increase in the fillers load as shown in Fig. 3.

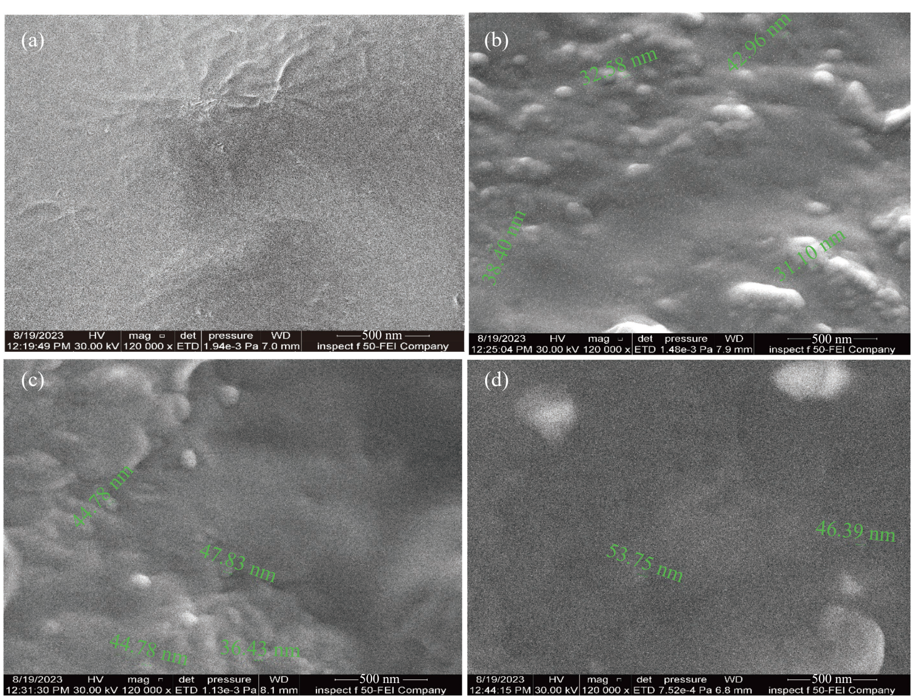

Fig. 2 FE-SEM images at 500× showing the materials surface of **(a)** G1, **(b)** G2, **(c)** G3, and **(d)** G4.

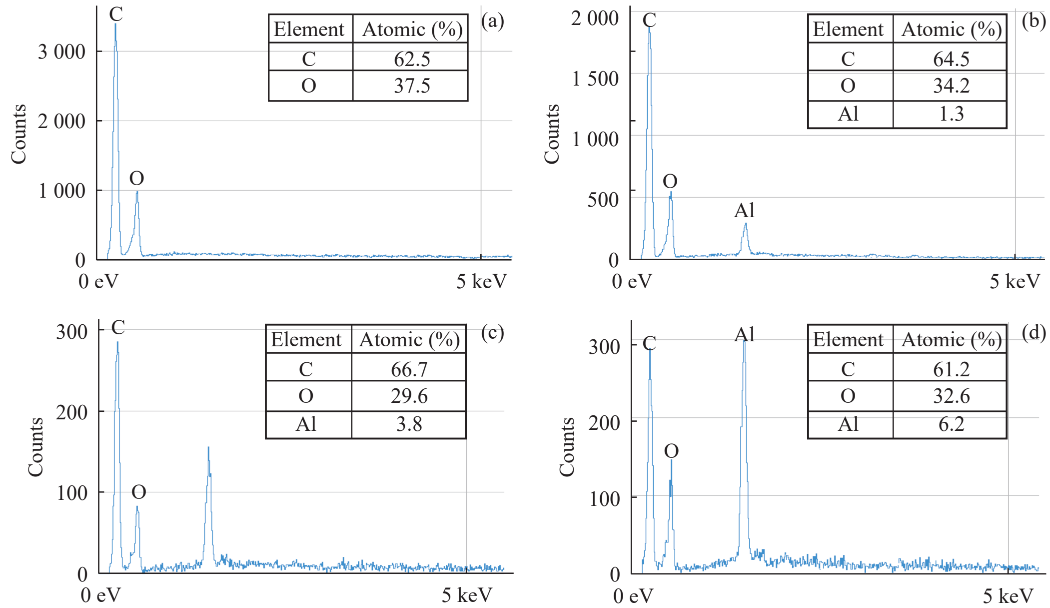

Fig. 3 EDX peaks of **(a)** G1, **(b)** G2, **(c)** G3, and **(d)** G4.

XRD

The diffraction peaks of the present nanocomposites are following the XRD pattern of Al_2O_3[27] and acrylic-based materials[28]. For Alumina, the diffraction peaks are located at (012), (104), (110), (113), (024), (116), (018), (024), (030), and (119). The broad peak at 2θ = 19° is assigned to acrylic-based materials. The obtained X-ray peaks do not contain any peaks parallel to any impurities that would suggest Al_2O_3 is in its single-crystalline phase (Fig. 4).

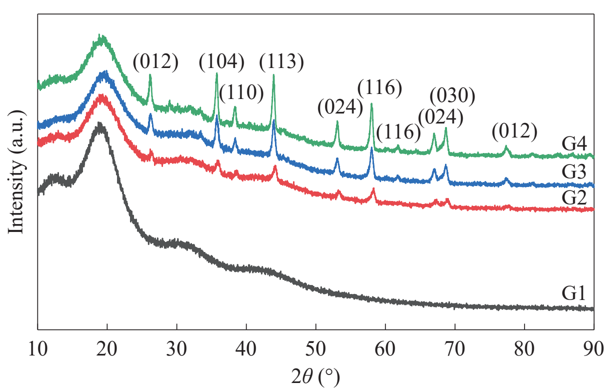

Fig. 4 XRD peaks of G1, G2, G3, and G4.

Glass transition temperature.

Figure 5 represents the mean value, SD, and Dunn's multiple comparison tests of the glass transition temperature. There is a highly significant increase ($p \leq 0.01$) in the T_g mean value as the percentage of alumina nanofillers increased. There is a highly significant difference, a significant difference, and a non-significant difference between G2, G4, and G3, and the control group G1.

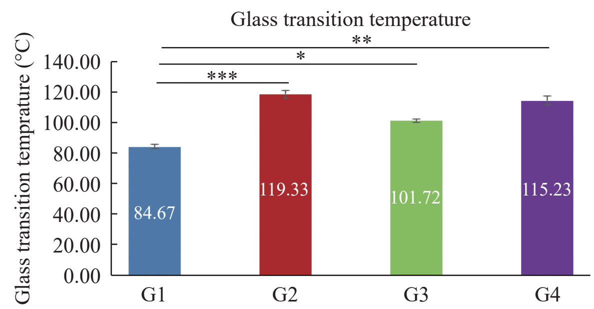

Fig. 5 Glass transition temperature of the experimental groups.

Coefficient of thermal expansion and contraction

Figure 6 represents the mean value, SD, and Dunn's multiple comparison tests of the thermal expansion properties at 30, 50, and 70 ºC.

There is a highly significant difference ($p \leq 0.01$) in the coefficient of thermal expansion and contraction between the experimental groups.

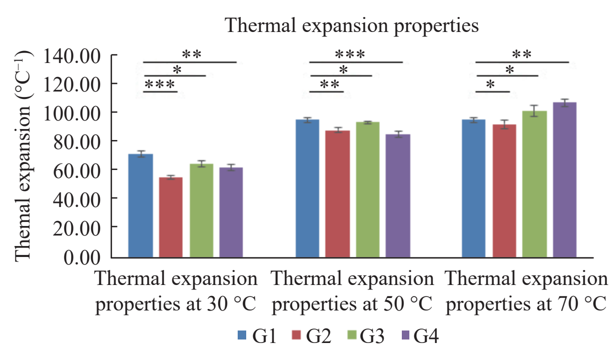

Fig. 6 Thermal expansion properties of the experimental groups.

However, a non-significant difference was found in G3 at all tested temperatures in comparison to G1. Additionally, there is a highly significant reduction at 30 ºC, a significant reduction at 50 ºC, and a non-significant reduction at 70 ºC between G2 and G1. For G4, there is a high significant reduction at 50 ºC and significant differences at 30 and 70 ºC, respectively in comparison to G1 (Fig. 6).

Modulus of elasticity test

Figure 7 represents the mean value, SD, and Dunn's multiple comparison tests of the modulus of elasticity at 30, 50, and 70 ºC.

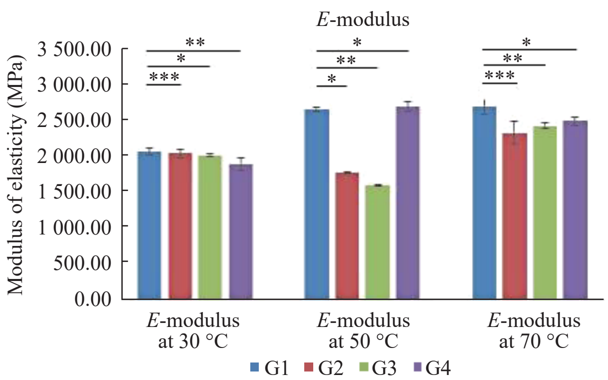

Fig. 7 Modulus of elasticity of the experimental groups.

There is a highly significant difference ($p \leq 0.01$) in the modulus of elasticity between the experimental groups. However, a non-significant difference was found in G4 at 50 and 70 ºC in comparison to G1, while a significant reduction at 30 ºC. On the contrary, a significant difference was found in G3 at 50 and 70 ºC in comparison to G1, while a non-significant difference at 30 ºC was noticed. A highly significant difference at 30 and 70 ºC was reported between G2 and G1. However, at 30 ºC, a non-significant difference in comparison to G1 was found (Fig. 7).

Discussion

Researchers are interested in polymer nanocomposite because of its unique features that are obtained from its two components. In this research, the effect of adding (Al_2O_3, nanofillers) to an acrylic soft liner was evaluated by studying the outcome of this process on glass transition temperature, coefficient of thermal expansion and contraction, and *E*-modulus of the acrylic soft liner. These types of nanofillers were selected because of their good thermal properties [29] in addition to their properties as nanofillers [14]. Theoretically, in a polymeric matrix, the nano-fillers are anticipated to scatter more uniformly than big micro-fillers, and this interaction will affect the characteristics of the composite materials [30]. However, this dispersion may be difficult due to the Van der Waals forces between the nanoparticles [31] . High-resolution field emission scanning electron microscopy was used to examine the morphology of the nanocomposite. Nearly smooth surfaces with linear lines that result from processing were seen in the FE-SEM of G1. As the filler load increased, aggregations were noticed as white fused particles (Fig. 2). This indicates the mixing technique was effective in breaking particle agglomerates until the filler load increased to 1.5 wt%. The O and C components are identified by the EDX analysis of G0–G4 as acrylic material, whereas the Al is identified for alumina nanopowder. When Al_2O_3 was added, and as the filler load increased, the Al and O contents were increased (Fig. 3). In Fig. 4, the XRD peaks of alumina are increased in intensity as the filler load was increased. The glass transition temperature of the resulting nanocomposite was higher than the control group. As T_g is dependent on the type and amount of nanofillers, T_g was increased as the filler load was increased from 0–1.5 wt%. Another explanation may be related to the melting point of alumina nanofillers which is considered higher than the melting point of the acrylic soft liner (Fig. 5).

The coefficient of thermal expansion and contraction and *E*-modulus are tested over a physiologic temperature range (20–70 ºC). Regarding the coefficient of thermal expansion and contraction, the introduction of (0.5, 1.0, and 1.5) wt% nanofillers into heat cure acrylic soft liner showed a reduction in its value at 30 and 50 ºC (Fig. 6) because the molecular mobility of the polymer was constrained by the matrix's higher interfacial contact with the nanofillers [32]. In addition, increasing the filler load can limit the mobility of macromolecule chains and improve thermal characteristics because of the homogeneous distribution of extremely fine size and high surface area in the nanofiller [33]. In other words, there was a decrease in the expansion and contraction by thermomechanical forces. In general, the value of *E*-modulus decreased when nanofillers were added to heat-cure acrylic soft liners as opposed to pure soft liners (Fig. 7). This decrease was brought on by more contact between the heat-cure acrylic soft liner molecules and the many nanofillers' surfaces [5].

Enhancing the soft denture liner properties may provide a chance to increase their effectiveness. For example, retrofitting implant parts can be managed during using soft liner retained implant supported dentures as it distribute the forces over the abutments, as a result, it may enhance the tissue health and the overall treatment success [34]. Additionally, the enhanced resiliency and handling properties of the soft denture liners may be adopted during the fabrication of palatal lift prosthesis [35].

Conclusion

Within the limitation of this study, the incorporation of 0.5 wt% of Al_2O_3 nanopowder into heat-polymerized soft denture lining material offers greater thermal and mechanical stability than un-modified heat polymerized soft liner material. Moreover, increasing the T_g of the nanocomposites can improve the handling properties and solubility.

CRediT Author Statement

Ban Saad Jasim: Conceptualization, investigation, and methodology. **Hasanain K.A. Alalwan**: Writing–original draft, supervision, and visualization. **Abdalbseet A. Fatalla**: Project administration, data curation, and formal analysis. **Manar E. Al-Samaray**: Data curation, writing–review, and editing.

Conflict of Interest

The authors have no conflicts of interest relevant to this article.

Supporting Information

The data used to support the findings of this study are available from the corresponding author upon request.

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