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Original article

Impact of chitosan modification on the material properties of acrylic resin base

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ABSTRACT

Background: *3D-printed polymethyl methacrylate is a light-cured commercial resin used in the 3D printing sector due to its affordability, good adaptability, minimal odor, and low irritation.* ***Purpose:*** *To investigate the impact of modified chitosan on the surface hardness and flexural strength of printed dental resin.* ***Methods:*** *A modified chitosan solution was cross-linked with adipic acid at concentrations of 0.1, 0.05, and 0.01 wt.% and then added to 3D-printable acrylic resin at 2, 5, and 10 wt.%. After addition, samples were prepared to test surface hardness and flexural strength. A total of 100 specimens were used in the research, grouped into 10 sets. Five specimens were prepared for each additive percentage, and five specimens served as a control group (3D-printable resin without modification) for each test.* ***Results:*** *The results showed that the (adipic acid/chitosan) 0.1/2 wt.% group had the highest flexural strength (134.370 MPa) and surface hardness (32.46 VHN), while the lowest flexural strength (49.198 MPa) and surface hardness (21.22 VHN) were observed in the (adipic acid/chitosan) 0.01/10 wt.% group.* ***Conclusion:*** *Modification of chitosan with adipic acid positively influences the flexural strength and surface hardness of 3D-printed denture bases. However, increasing the chitosan content beyond 2 wt.% reduces both surface hardness and flexural strength in modified 3D-printed polymers.*

Keywords: *3D acrylic resin; adipic acid; chitosan; dicarboxylic acid; flexural strength; hardness; medicine*
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INTRODUCTION

Polymethyl methacrylate has mechanical characteristics that limit its use in biomedicine.[1] Most milled denture-base materials, as well as thermally polymerized resin, have better mechanical properties than 3D-printed denture bases.[2,3]

The simplest method to classify 3D printing materials is based on their state during processing in the 3D printer. Liquid resin-based materials are among the most widely used in digital dentistry and are typically processed using stereolithography (SLA), digital light processing (DLP), and PolyJet printing (PJP) techniques. Some materials are also used in powder form. Binder jetting, selective laser sintering (SLS), and Multi Jet Fusion (MJF) use polymer powders to produce sections. Direct metal laser sintering and selective laser melting utilize metal powders to create parts. Fused deposition modeling employs solid-based materials in the form of polymer filaments for 3D printing.[4,5]

The term "light curing" refers to a broad category of 3D printing technologies that use photosensitive resins, which cure upon exposure to light.[6] Photo-curable resins are the most commonly used 3D-printable materials for dental applications. SLA technology cures liquid resin with a scanning laser to produce items layer by layer. DLP technology, on the other hand, uses light-emitting diode projectors to cure the resin. Liquid resin materials processed in DLP or SLA are used to create dental models, surgical guides, custom impression trays, temporary and permanent crowns, and gingiva masks. A recent example of a desktop-based low-force stereolithography printer for dental applications is the Form 3D series by Formlabs (Somerville, Massachusetts, USA), which offers a wide range of materials for various dental applications. Notable original equipment manufacturers of DLP-based 3D printers for dental applications include NextDent (Soesterberg, the Netherlands), SprintRay (Los Angeles,

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California, USA), Asiga (Alexandria, New South Wales, Australia), EnvisionTEC (Dearborn, Michigan, USA), Rapid Shape (Heimsheim, Germany), and DWS (Thiene, Vicenza, Italy).[7]

PJP is a commonly used technique that applies liquid waxes and resins to 3D print objects. PJP is used for anatomical models and implant drill guides, and some flexible materials can be used to produce orthodontic splints. However, the materials, equipment, and operating costs of PJP technology are higher than those of other 3D printing techniques.[4] Chitin is the main structural component of crustacean shells and arthropods. Chitosan has demonstrated antifungal properties, particularly against *Candida albicans*, both in its unbound polymer form and as a derivative.[8,9] The use of 3D printing in the design and fabrication of complete dentures is currently being investigated. Tissue adaptation is crucial for masticatory performance, retention, and the stability of the record base in complete and removable dentures. Additionally, manufacturing methods may influence the performance of the denture base.[2,3]

To enhance the characteristics of chitosan and expand its applications, various methods have been used, including cross-linking, graft copolymerization, chemical modification, and blending.[10,11] The selection of appropriate cross-linkers is crucial for transforming chitosan's macromolecule into a highly strengthened and biocompatible material. Chitosan polysaccharide is converted into a homogeneous solution through mutual cross-linking between dicarboxylic acids (DCAs) and chitosan in a water medium. The biomaterial's strength is further enhanced by common ionic interactions.[12]

The current work investigates the dual function of DCAs when combined with chitosan. Additionally, a wet lab study (preparation of 3D biomaterials in vitro) was conducted to support the hypothesis. The results demonstrate that adipic acid is an ideal compound for biomaterial preparation.

MATERIALS AND METHODS

The research was conducted in vitro and approved by the College of Dentistry, University of Mosul. The study was approved by the Research Ethics Committee, reference number UOM. Dent. 23/38. Chitosan (shrimp source, China) solution (10 g/L) was prepared by dissolving 5 g of chitosan in 500 ml of acetic acid (Merck, Darmstadt, Germany). The solution was then stirred, heated at 60°C, and filtered to remove dust and other impurities. Air bubbles were eliminated by maintaining the solution at 20°C for two hours.[13]

Modified chitosan solutions (0.1 g/L, 0.05 g/L, and 0.01 g/L) were prepared by dissolving 1 g of chitosan in 100 ml, 50 ml, and 10 ml of adipic acid (Sigma-Aldrich, USA), followed by gentle stirring and heating at 55°C. Air bubbles were removed by holding the solutions at 20°C for two hours.[13] To prepare modified chitosan acrylic solutions, adipic acid (0.1, 0.05, and 0.01 g/L) was added to 3D-printable acrylic resin (DATABASE 3D+ denture base material, Asiga, Australia) at concentrations of 2 wt.%, 5 wt.%, and 10 wt.%.

The designs were saved as STL files. Base and support structures were added to the 3D-designed samples, and the slicing and printer settings were configured. The file was then exported to the printer, a commercial 3D printer (Asiga, Australia).[14]

The thickness of the slices used was 50 µm in the Z-axis, and the curing time was seven seconds per slice, following the guidelines provided by the manufacturer. Each group of samples with its respective concentrations was printed together on the same plate, using the same printing cycle, and under the same room temperature conditions to avoid differences in conditions and ensure standardization for all samples. After printing was completed, the samples were moved to the washing and curing machine (Asiga, Australia) in accordance with the material manufacturer's instructions.

The samples were washed for four minutes inside the spinning machine using 99% isopropyl alcohol to eliminate unpolymerized resin and reduce the residual monomer remaining on the surface of the samples. Excess material was carefully removed with a sharp blade, and the samples were then checked for any defects. The specimens were dried with moderate air pressure for 30 seconds before being returned to the device for curing. The curing cycle was completed under blue light with a wavelength of 405 nm, and the curing table was rotated for 45 minutes.[15]

After the addition processes, the samples were prepared for surface hardness and flexural strength tests. The total number of specimens used in this research was approximately 100. Five specimens were made for each percentage of each additive material added to the printable resin, totaling 90 specimens, with an additional 10 specimens for the control group (3D printable resin without any addition or modification). Flexural strength was considered the primary mode of failure, as enhancing the flexural strength of dental devices, particularly denture bases, is crucial. These devices are subjected to forces that can lead to fractures, especially during use.[16] For the modified materials, the flexural strength was determined using the three-point bending test in accordance with ISO standards. The samples were exposed to compressive load until fracture at a crosshead speed of 5.0 mm/min, using a Universal Testing Machine (Electro-Plus TM E3000; Instron, Buckinghamshire, UK). Flexural strength was calculated using the following formula:[17]

$$\text{Flexural strength} = 3FL/2bh^2$$

Where (F) is the fracture load in (N), (L) is the distance between the two supports, (b) is the width of the specimen, and (h) is the thickness of the specimen.

Surface hardness test: In this test, 50 specimens were prepared, with five specimens for each group used in the study. The specimens of each group were made with the

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following dimensions: 10 x 10 x 3.3 ± 0.2 mm (length, width, height, respectively). Five measurements were recorded from different areas of each sample, and the mean value was then calculated.[18] The hardness of the samples was measured using a Micro-hardness Vickers tester (HV-1000A, Korea), designed for hard materials. It was equipped with an indenter in the form of a round steel ball with a diameter of 1.25 mm.[19] The sample was placed on the solid plane of the apparatus, with the needle positioned 12 mm from the specimen's edge. The samples were subjected to a fixed minor load of 44.5 N. The Micro-hardness Vickers tester measured the indenter's relative movement after each indentation, and the equipment automatically converted this measurement to a scale graduated from 0 to 100 units. The final hardness value was determined by visually reading the analog scale after the application of the load within one second.

RESULTS

The results showed a decrease in the flexural strength of the 0.01/10%, 0.05/10%, and 0.1/10% groups, respectively, compared to the other groups in the study. These groups failed to meet the lower limit of the ISO requirement for flexural strength, which is 65 MPa. The highest value of flexural strength (134.4 ± 2.5 MPa) was achieved by the 0.1/2% group, while the lowest value (49.2 ± 1.7 MPa) was achieved by the 0.01/10% group. The Duncan multiple range test found a statistically significant difference at $p < 0.05$ in the flexural strength tests of all groups used in this study, except between the 0.01/5% and 0.05/5% groups, and among the 0.01/10%, 0.05/2%, and 0.1/10% groups. The results also demonstrated a decrease in surface hardness in the 0.01/10%, 0.05/10%, and 0.1/10% groups, respectively (Figure 1).

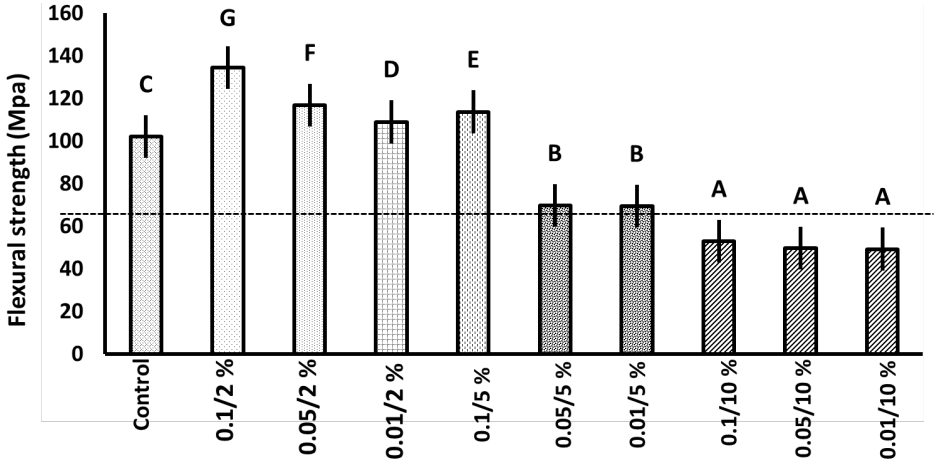

Figure 1. Flexural strength test of the studied groups. Data are expressed as mean ± SD. The Duncan multiple range test was used to compare subcategories for significant differences at $p < 0.05$. The dashed line represents the ISO flexural strength requirement (65 MPa). Similar letters represent non-significant differences ($p > 0.05$), while different letters represent significant differences ($p < 0.05$).

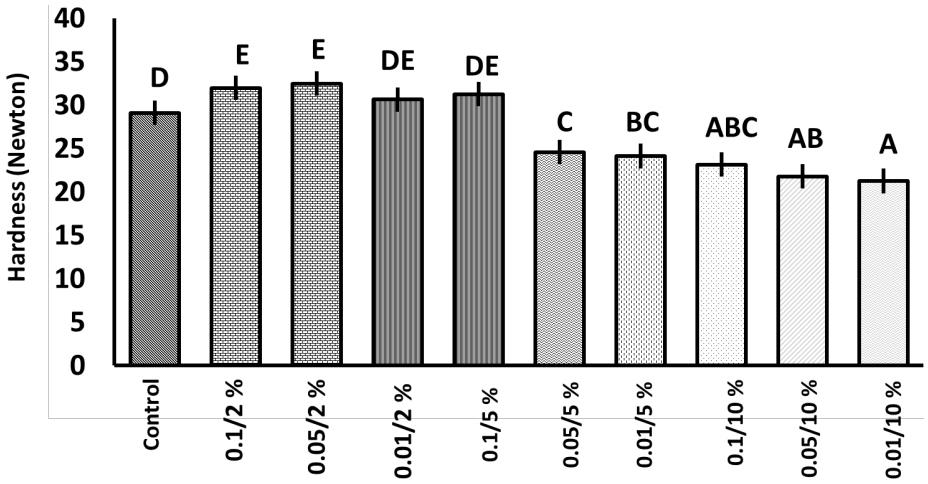

Figure 2. Hardness test of the studied groups. Data are expressed as mean ± SD. The Duncan multiple range test was used to compare subcategories for significant differences at $p < 0.05$. Similar letters represent non-significant differences ($p > 0.05$), while different letters represent significant differences ($p < 0.05$).

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The highest value for surface hardness (32.5 ± 1.6) was achieved by the 0.1/2% group, while the lowest value (21.2 ± 0.8) was achieved by the 0.01/10% group. The Duncan multiple range test determined a statistically significant variation at $p < 0.05$ in the hardness test between the control group and all other groups used in the study, except for the 0.1/5 wt.% and 0.01/2 wt.% groups. Additionally, a statistically significant variation at $p < 0.05$ was observed in the hardness test of the 0.1/2% group compared to all other groups in the study, except for the 0.05/2%, 0.1/5%, and 0.01/2% groups (Figure 2).

DISCUSSION

This investigation demonstrated the efficacy of adding modified chitosan with adipic acid on the flexural strength of printed dental resin (Asiga). Additionally, a comparison of this property was made between the modified groups and the control 3D-printed denture resins. According to the results, modified chitosan had a notable impact on the surface hardness and flexural strength of the 3D-printed resin material. As a result, the hypothesis was accepted.

The printing technology most commonly used for dental applications is DLP due to its superior resolution, rapid processing speed, and affordable printer and component costs.[20,21] The highest bending force a material can withstand before yielding is known as its flexural strength. Denture bases are prone to fracture when exposed to static or dynamic loading.[22]

Surface hardness of the specimen represents the strength of the material's surface and serves as an indicator of abrasion resistance. Low hardness may lead to increased scratches, resin surface damage, and dimensional changes during mechanical brushing of the dentures or when chewing hard food. Therefore, the properties of the printed denture base should be evaluated for clinical application. In this study, the modification of the printing control material was aimed at enhancing and improving the surface properties.

The results of this investigation demonstrated the impact of the modification on the surface characteristics of 3D printed resin using a more efficient technique that could enhance the properties of dentures. Therefore, the denture would undergo prolonged polymerization, which improves the degree of conversion and enhances the physical-mechanical qualities.[20]

The printed dental resin exhibited lower surface properties compared to the heat-cured resin.[23,24] As a result, the 3D-printed resin had lower values for flexural strength and surface hardness, which could be attributed to the combination of curing conditions and the reactivity of the monomers in the 3D-printing resin. This led to a lower degree of double-bond conversion compared to traditional acrylic resins.[22,25] Another factor contributing to the reduced mechanical qualities might be the weakness in the inter-layer bonding between the printed layers.[23,26]

Nevertheless, the printed dental resin material examined in the research met the ISO standards for flexural strength (65 MPa).[27] As a result, 3D-printed materials can be considered for use in making denture bases. According to the findings of this study, the group modified with chitosan and adipic acid (0.1/2 wt.%) exhibited the highest values for both flexural strength and hardness. The ANOVA test revealed significant differences among all the examined groups. This can be attributed to the fact that biomaterials derived from chitosan have been identified for various biomedical applications. However, the use of cross-linkers has restricted their applications, and these results align with previous studies.[28,29]

These findings suggest the necessity for appropriate cross-linkers to modify chitosan macromolecules, supporting studies that indicate cross-linkers can transform chitosan into a biocompatible, high-strength, porous material. DCAs can interact with the -NH2 group in chitosan at various temperatures without requiring activation of the -COOH group of the DCA. This study explored the potential for a reaction between DCAs and chitosan polysaccharides.[12] DCAs are an optimal choice, as they do not require high temperatures to initiate the reaction, thus eliminating the need to stimulate -COOH groups, which could otherwise hinder solvent usage. Adipic acid, in particular, is sparingly soluble in water at room temperature.[30]

Furthermore, it was assumed that the bi-functional action of the DCA played two roles during its interaction with the chitosan molecule.[12] The cooperative cross-linking of DCAs with chitosan in water transformed the chitosan polysaccharides into a solution, and the resulting ionic interactions enhanced the strength of the modified material.

The results also revealed that modifying the acrylic resin with chitosan particles up to 2% had significant positive effects on the flexural strength and surface hardness of the acrylic resin. However, the addition of chitosan at 5% and 10% resulted in substantial adverse effects on the mechanical characteristics of the printed acrylic resin.

As the percentage of chitosan increased from 2% to 10%, there was a decrease in both surface hardness and flexural strength. This could be due to the chitosan particles functioning as an impurity in the poly(methyl methacrylate) matrix, thereby reducing both properties in the acrylic resins.[31]

Chitosan may also negatively affect the degree of conversion during polymerization, leading to an increase in the quantity of residual monomer, which acts as a plasticizer. Additionally, agglomeration of chitosan particles is possible, and these agglomerated particles could serve as stress concentration points within the acrylic resin matrix. Overall, these factors contribute to the decrease in both hardness and flexural strength.[32,33]

Resin materials absorb water in a diffusion-controlled manner, either through specific chemical interactions or by penetrating spaces such as micro-voids.[34–36] The extent of water absorption depends on the polarity of the resin and

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the availability of sites for hydrogen bonding with water. The interaction between water and the polymer chain can lead to slight chemical breakdown, resulting in a decrease in the material's strength.[37]

Similar to conventional materials, the printed resin exhibited a tendency for water sorption. However, in contrast to the heat-cured material, its water solubility was higher. This could be attributed to the fact that polymers in heat-cured resin are polymerized for a longer period at a higher temperature, which results in reduced water sorption, solubility, and monomer residue concentration, as indicated previously.[38–40] Additionally, variations in the composition of the printed and modified resin materials should be considered, as the chemical composition of the resin material affects both its solubility and water sorption.[41,42]

The results of this study suggest that clinicians should consider the differences in mechanical characteristics between modified and 3D-printed materials used for constructing denture bases.[43] Further improvements in the properties of 3D-printed resin materials through composition modification or reinforcement are still needed. The correct selection of post-curing methods could also serve as an option for enhancing these materials. This investigation was limited to studying only one type of printed dental resin, specifically the material where dicarboxylic acid was used to modify chitosan before adding it to the resin at three different percentages. Further research into other 3D-printed materials and post-curing techniques is recommended.

In summary, this study concluded that the modification of chitosan with adipic acid had a positive impact on the flexural strength and surface hardness of the modified printed dental resin. Increasing the percentage or volume of adipic acid from 0.01 or 0.05 to 0.1 g/L led to an improvement in the flexural strength and surface hardness of the printable acrylic resin, potentially enhancing the flexural properties of resins used in dental appliances. However, an increase in the percentage of chitosan used to modify the 3D-printed polymers resulted in reduced flexural strength and surface hardness.

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