Addition of Glass Fibers and Titanium Dioxide Nanoparticles to the Acrylic Resin Denture Base Material: Comparative Study with the Conventional and High Impact Types

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Abstract

Purpose: The aim of this study was to clarify the effect of addition of glass fibers and titanium dioxide nanoparticles to the conventional acrylic resin. The tested parameters were monomer release, deflection at fracture, flexural strength, flexural modulus, and toughness. The modified acrylic resin groups were compared to the conventional unmodified and high impact types. The correlation between the tested material properties was also evaluated.

Materials and methods: The materials used were conventional unmodified and high impact acrylic resins. The conventional acrylic resin was modified using 5% glass fibers and 5% titanium dioxide nanoparticles. Specimens were prepared according to the manufacturer's instructions and American Dental Association Specification No. 12. Monomer release was measured using isocratic high-performance liquid chromatography. Deflection at fracture, flexural strength, and flexural modulus were measured using three point-bending test with a universal testing machine. The toughness was related to the total area under the load-deflection curve up to the breaking point. The correlation between the tested properties was clarified.

Results: All materials released monomer with varying values. The tested materials exhibited comparable values of deflection at fracture. Specimens modified with glass-fibers showed improved flexural strength and toughness similar to that of the high impact acrylic resin. Specimens modified with titanium dioxide nanoparticles exhibited reduction in the flexural properties and toughness. No significant changes were observed in the flexural modulus. There were positive correlations between the flexural strength, flexural modulus and toughness. On the contrary, there was negative correlation between deflection at fracture and flexural modulus.

Conclusions: The most commercially successful method for reinforcement to date is the rubber toughening. The conventional acrylic resin denture base material could be reinforced by glass fibers while titanium dioxide nanoparticles could not. The tested materials released comparable amounts of monomer.

Key Words: Acrylic Resin, Glass Fibers, Titanium Dioxide, Nanoparticles, Mechanical Properties, Monomer Release.

Introduction

To date the most commonly used denture base materials are the conventional heat-cured acrylic resins. The popularity of acrylic resin depends on its ease in manipulation, ease in finishing and polishing, as well as it needs inexpensive equipments. The acrylic resin is stable in the oral conditions and has high aesthetic quality. Till now the acrylic resin denture base material does not fulfill all the requirements of acceptable mechanical properties [1].

Polymerization of PMMA by heat may be affected by a variety of time and temperature parameters, and varying amounts of unreacted methyl methacrylate monomer remain after the curing cycle. All acrylic resins contain variable residual monomer levels depending on the ambient conditions and the efficiency of heat transfer [2]. Many investigations have demonstrated that residual monomer content varied considerably with curing conditions, and the amount of residual monomer was one of the primary factors affecting the properties of the denture base materials produced under different curing cycles [3-5]. The residual monomer may diffuse from acrylic, resulting in an irritation or allergic side effects. Proper processing techniques minimize residual monomer content in denture bases and keep residual monomer in the range of 1-3% that is well tolerated by most individuals [6].

An acceptable acrylic polymerization method is the one which is capable of achieving the best properties of the acrylic resin, e.g.

fracture toughness, static strength, flexural modulus and monomer release. It has been shown that high levels of residual monomer are adversely affect acrylic resin properties like hardness and porosity [7,8]. One of the properties of acrylates is the monomer release, which causes dimensional instability, thereby subjecting the material to internal stresses which might result in crack formation and, eventually, fracture of the denture [9,10].

The acrylic resin materials are typically low in strength, soft and fairly flexible, as well as brittle on impact, and fairly resistant to fatigue [11,12]. As the fracture resistance of a denture base resin is important, many approaches have been used to strengthen acrylic resin dentures. These approaches involve the use of metal wires or plates, fibers, metal powder or rubber toughening agents. These reinforcing methods were done to improve the mechanical properties of the denture base resins and to overcome the problem of denture fractures. The fibers that have been used to reinforce denture base resins were aramide fibers, carbon / graphite fibers, polyethylene fibers and glass fibers [11-14].

Therefore, it is important to know the mechanical properties of high impact acrylic resins through the addition of cross-linking agents and rubber incorporation in acrylic resin. The additives could affect the toughness, microstructure and deformation behavior under the impact and flexural tests [12]. The methods used to improve the inherent properties of polymethylmethacrylate have included using alternate polymers such as polycarbonate, nylon, co-polymers and

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reinforcing agents [15].The most commercially successful method of reinforcement to date is rubber toughening; however, such materials have compromised flexural properties. A rubber phase may be introduced into the acrylic matrix as either co-polymer, rubber particles or as core-shell particles. The polymers are reinforced with butadiene-styrene rubber. The rubber particles are grafted to methyl methacrylate to bond to the acrylic matrix [6,12].

In evaluation of denture plastics, the transverse strength measurements are used to a greater extent than either tensile or compressive strength, because this test more cl g agent [(C10 H20 O5 Si), Promochem G osely represents the type of loading *in vivo*. Because the geometry of the denture base is complex and stresses can be concentrated in flaws on the surface or in frenum notches, cracks might occur in the denture base. Fatigue fracture of denture bases is common clinical problem due to its brittleness and continuous exposure to cyclic loading [6]. The aim of this study was to clarify the effect of addition of glass fibers and titanium dioxide nanoparticles on monomer release, deflection at fracture, flexural strength, flexural modulus, and toughness of acrylic resin denture base material. The correlation between the tested properties was to be evaluated.

Materials and Methods

The materials used were, Acrostone conventional heat-cured acrylic resin (Acrostone, WHN, England) and high impact type (Metrocryl Hi, Metrodent, LTD, England). Dental stone (Hydrocal Dental stone, Moldano, Bayer Lerekusen, and Germany) and polyvinyl siloxane impression material (Aquasil, DENTSPLY International Inc, York, PA, USA). Glass fibers (Gulf glass fibers, Tech. Ind. Saudi Arabia) and titanium dioxide nanoparticles (TiO$_2$) powder ((Batch number: MKBC-4174, Sigma–Aldrich). Glass fibers of 10 μm in thickness and 1 mm in length and titanium dioxide of 21nm particle size were used. Silane coupling agent [(C$_{10}$ H$_{20}$ O$_5$ Si), Promochem GmbH Postfach Wesel, Germany].

Sample preparation

Specimens were prepared according to the manufacturer's instructions and American Dental Association Specification No.12. [16]. Specimens were packed and processed in dental stone molds which were replicated from polyvinyl siloxane impressions using stainless steel molds. Baseplate wax was melted and poured into the stone molds, which were then flasked in dental stone as usual [17]. The acrylic resin was proportioned and mixed according to the manufacturer's instructions. Four groups were prepared as follows:

Group I: Control unmodified group (prepared from the conventional heat-cured acrylic resin).

Group II: Glass-fiber modified group (acrylic resin powder mixed with short glass fibers 5% by weight).

Group III: High impact acrylic resin, ready-made polymer modified with butadiene styrene rubber.

Group IV: Titanium dioxide nanoparticles modified group (acrylic resin powder mixed with titanium dioxide 5% by weight).

The glass fibers were treated by Silane coupling agent to wet the fibers in an attempt to improve the bonding of the fibers with acrylic resin. A solution of 0.3 g silane coupling agent in 100 g of a mixed solution of water and alcohol was used to treat 30 g of glass fibers. The fibers were dipped into the solution for 60 minutes. The fibers were then allowed to precipitate by separation and left to dry at an ambient temperature. This was followed by heating at

110°C to 120°C for 5-10 minutes according to the manufacturer, s recommendations. The treated fibers were then added to the heat-cured acrylic resin powder in 5% by weight. The fibers were mixed thoroughly to be dispersed into the resin powder. Titanium dioxide (TiO$_2$) nanoparticles were mixed thoroughly to the acrylic powder by hand before mixing with the acrylic monomer.

The proportioned polymer/monomer ratios were mixed, on reaching dough stage; the mixture was kneaded and packed into the prepared molds. Trial closure was performed with a hydro press at 40,000 N [18].The flask was clamped, and low pressure was maintained for 30 minutes to allow proper penetration of monomer into the polymer, even flow of the material, and outward flow of the excess material. The material was processed according to the manufacturer's instructions. The flask was immersed in a water bath at room temperature in a thermostatically controlled unit. The temperature was raised slowly to 73 ± 1°C for 90 minutes, and then elevated to the boiling point at 100°C for 30 minutes. The flasks were allowed to bench cool before opening. The cured plates were carefully removed from the molds, the excess flush was removed and the specimens were finished using polishing machine with wet water proof silicon carbide paper disk 600 grit size.

Monomer release testing

A total of 28 disc-shaped specimens were fabricated, seven specimens for each material. The specimen's dimensions were 50 mm diameter and 3 mm thickness. Specimens were tested for monomer release according to the ISO 1567:2000. Each specimen was stored in a sealed container, which held 10 ml of deionized water and was kept in dark conditions at 37°C for 7 days. At the end of the leaching period, the sample was removed, and the amount of Methylmethacrylate (MMA) was determined. Three water extractions with 0.6 ml of n-hexane (Merck, Darmstadt, Germany) were used. The final volume of extract was adjusted to 2.0 ml with n-hexane. The concentrations of released MMA were measured using isocratic high-performance liquid chromatography (HPLC; Waters, Milford, MA) with a reverse-phase column (LiChroCART 250-4 Cartridge, LiChrospher 100 RP-8 (μm) column, Merck. Acetonitrile (HPLC grade, Merck) with water (50:50) was the mobile phase; the flow rate was 1 ml/min, with detection at 254 nm. The peak area of MMA was calculated on the HPLC trace. Each specimen was measured three times. The amount of leached MMA was determined using a standard calibration curve with MMA concentrations of 1,5,10,100,500 & 1000 ppm as controls [19].

Flexural properties testing

A total of 40 bar-shaped specimens were prepared, ten specimens for each material. The specimen's dimensions were 65 length, 10 width and 2.5 mm thickness. Specimens were stored in water at room temperature for 24 hours before testing. Specimens were tested for flexural strength using a three point-bending test with a universal testing machine (MTS systems, ADAMEL, LHOMARGY, DY 25) at a crosshead speed of 2-mm/ min [20]. A load was applied by a centrally located rod until fracture occurred. The chart recorder on the testing machine produced a complete force versus deflection history of each test. The force and deflection at fracture were obtained from the data of the machine. The flexural strength was calculated in Mega- Pascal (Mpa) from the following formula [12,18,21]:

$$S = \frac{3FL}{2bd^2}$$

Where S=flexural strength, F=load at fracture (N), L=distance

between supports (mm), b=width of sample (mm), and d=thickness of sample (mm).

The flexural modulus was calculated in Gpa from the following formula: [12,18,21].

$$E = \frac{FL^3}{4Ybd^3}$$

Where E=flexural modulus, F=load at P (N), L=distance between supports (mm), Y=deflection at P (mm), b=width of sample (mm), and d=thickness of sample (mm).

Toughness testing

A total of 40 bar-shaped specimens were prepared, ten samples for each material with the previous dimensions.The testing machine was computer-interfaced to process the data with a specifically designed program. Specimen's thickness and width data were introduced into the computer. The testing machine was conducted the destructive test and measured the breakage load and the beam deflection. The process was visually observed on a monitor. Toughness is related to the total area under the load-deflection curve up to the breaking point and represents the energy absorption needed to break a specimen [22,23]. Toughness was recorded directly from the computer of the testing machine in N-mm.

Results

The mean values of monomer release are presented in *Table 1*. One-way ANOVA showed a significant difference among the tested materials ((P ≤ 0.001). LSD test showed no significant difference in monomer release between the control unmodified, fiber-modified and TiO_2 modified resins (P ≥ 0.05). The statistical analysis of the results revealed significant difference between the TiO_2- modified, fiber- modified and high impact groups (P ≤ 0.05). The high impact resin exhibited the lowest monomer release. There was a significant difference between the fiber-modified and high impact groups (P ≤ 0.05).

The mean deflection values at fracture are presented in *Table 2*. Statistically there was a significant difference among the tested groups (P ≤ 0.05). LSD test showed no significant difference between the unmodified group and the other treated groups (P ≥ 0.05). Also, there was no significant difference between high impact and fiber-modified types or between fiber-modified and TiO_2-modified materials. There was a significant difference found between TiO_2-modified and the high impact acrylic resin (P ≤ 0.05).

The mean flexural strength values are presented in *Table 3*. Statistically, there was a significant difference found among the tested groups (P ≤ 0.01). The statistical analysis showed that the flexural strength of the fiber-modified and high impactgroups was significantly higher than that of the unmodified and TiO_2-modified resins (P ≤ 0.001). There was no significant difference found between the fiber-modified and the high impact acrylic resins or between the unmodified and the TiO_2-modified types (P ≥ 0.05).

The mean values of flexural modulus are presented in *Table 4*. The statistical analysis of the results revealed no significant differences in the flexural modulus of the tested materials (P ≥ 0.05).

The mean values of toughness are presented in *Table 5*. Statistically, there was a significant difference among the tested groups (P ≤ 0.05). The toughness of TiO_2-modified acrylic resin was significantly the lowest when compared to the fiber-reinforced and high impact resins (P ≤ 0.05), whereas the unmodified and TiO_2-modified groups were not significantly different (P ≥ 0.05). There was no significant difference between the high impact and the fiber-modified resins (P ≥ 0.05).

There was a high positive correlation between flexural strength, flexural modulus and fracture toughness. There was a high negative correlation between the deflection at fracture and flexural modulus (*Table 6*).

Discussion

Acrylic resin denture fractures are still considered a major problem and several attempts have been made to increase the mechanical

Table 1. Mean and standard deviation of monomer release (ppm) of different acrylic resin denture base materials.

Materials	Mean ± SD	F-value	P-value	LSD
Unmodified acrylic resin	7.4(AB) ± 0.9	16.86	0.0008**	1.3857
High impact acrylic resin	4.3(C) ± 0.4			
Fiber-modified acrylic resin	6.5(B) ± 1.0			
Titanium dioxide	8.4(A) ± 0.5			
modified acrylic resin				

*p ≤ 0.001=highly significant.
Means with the same superscripted letters are not significantly different.

Table 2. Mean and standard deviation of deflection at fracture (mm) of different acrylic resin denture base materials.

Materials	Mean ± SD	F-value	P-value	LSD
Unmodified acrylic resin	6.0(AB) ± 1.2	2.6	0.05*	1.24
High impact acrylic resin	6.4(A) ± 1.5			
Fiber-modified acrylic resin	6.0(A,B) ± 1.9			
Titanium dioxide	5.4(B) ± 1.1			

*p ≤ 0.05=significant difference.
Means with the same superscripted letters are not significantly different.

Table 3. Mean and standard deviation of flexural strength (Mpa) of different acrylic resin denture base materials.

Materials	Mean ± SD	F-value	P-value	LSD
Unmodified acrylic resin	128.7(B) ± 19.1	5.3	0.004**	19.4
High impact acrylic resin	147.6(A) ± 22.9			
Fiber-modified acrylic resin	139.6(A) ± 24.9			
Titanium dioxide	113.5(B) ± 16.9			

** p≤ 0.01 = high significant difference.
Means with the same superscripted letters are not significantly different.

Table 4. Mean and standard deviation of flexural modulus (Gpa) of different acrylic resin denture base materials.

Materials	Mean ± SD	F-value	P-value	LSD
Unmodified acrylic resin	3.6(A) ± 0.9	0.49	0.7+	0.77
High impact acrylic resin	3.9(A) ± 0.8			
Fiber-modified acrylic resin	3.9(A) ± 0.8			
Titanium dioxide	3.5(A) ± 0.9			

+ P ≥ 0.05 = no significant difference.
Means with the same superscripted letters are not significantly different.

Table 5. Mean and standard deviation of toughness (N-mm) of different acrylic resin denture base materials.

Materials	Mean ± SD	F-value	P-value	LSD
Unmodified acrylic resin	47.5(B) ± 7.4	2.73	0.05*	7.8
High impact acrylic resin	56.8(A) ± 7.4			
Fiber-modified acrylic resin	53.7(A) ± 7.9			
Titanium dioxide	43.7(B) ± 11.1			

* P ≥ 0.05 = no significant difference.
Means with the same superscripted letters are not significantly different.

Table 6. Correlations between the studied mechanical properties.

	Deflection at	F-value	P-value	LSD
fracture	Flexural strength	Flexural modulus	Toughness	7.8
Deflection at Fracture	1	.008		
.959	-.655*			
.000	.063			
. 700				
Flexural Strength	-----	1	.483**	
.002	.816**			
.000				
Flexural Modulus	-----	-----	1	.466**
.002				
Toughness	-----	-----	-----	1

Correlation is significant at the 0.01 level (2-tailed).
** High positive correlations.
-* High negative correlations.

properties of the denture base resins. The fracture of the acrylic resin dentures is probably caused by multiple factors rather than the intrinsic properties of the denture base material [12]. The transverse (flexural) strength of a material is a measure of stiffness and the resistance to fracture. Flexural strength tests were considered more relevant to the loading characteristics of a denture base in a clinical situation [24].

Short fibers were used in this study because the short fibers represented a suitable size for manipulation and incorporation into the acrylic resin dough. The incorporation of milled glass fiber (1.2 μm in diameter, 0.8 mm in length and 2.7 g/cm³ in density) at various concentrations into Polymethylmethacrylate (PMMA) increases the wear resistance considerably but after a certain concentration it has no significant importance [25].

Free radical polymerization reactions are rarely proceeding to completion, leading to the presence of unreacted residual monomer in the polymeric material which affects the polymer properties. Therefore, complete conversion of monomer is the main goal of acrylic resin polymerization methods. A denture base material without or with very low residual monomer would therefore have a special commercial requirement [26].

During the polymerization process the amount of residual monomer decreases rapidly at first, then more slowly. The amount of residual monomer in a denture plastic processed at 70°C and at 100°C depends on the time of processing. If heat-processed materials are to be used for patients sensitive to residual monomer, processing for longer times in boiling water should reduce the monomer to an acceptable level. Because there is evidence that PMMA monomer has poor biocompatibility, every effort should be made to eliminate residual monomer or reduce it to very low levels [6].

The recently introduced denture base materials have lower residual monomer content. If the samples were immersed in water at 37°C for 24 hours, considerably more monomer was removed than during immersion in water at 22°C. So, it is recommended that the denture should be immersed in water at 37°C for one day prior to insertion. The monomer release reached its lowest level during 2-day soaking of varnished samples [27].

The residual monomer released from the control unmodified and the TiO_2-modified groups was greater than that of the others. Such a finding may be explained by the low-level degree of conversion. On the contrary, the amount of monomer released from the high impact group was the lowest. TiO_2-modified group released greater monomer than that of the fiber- modified and the high impact groups. Some authors concluded that the microwave cured acrylic resin with glass fibers have significantly lower residual monomer content from the 1st day of immersion when compared to that of heat cured acrylic resin (with and without fibers) which shows the same level of residual monomer at the 7th day of immersion [28].

The results of this study indicated that the deformation of the tested materials was quite similar. The data obtained were in accordance with the British Standard Specification 1989 (BS 2487) for denture base resins which specified transverse deformation limits that are from 1 to 2.5 mm for a force of 15-35 N and 2-5 mm for a force of 15-50 N. The average breaking force of acrylic resin should not be less than 55 N [24].

The transverse strength of a material is a combination of compression, tensile and shears strengths. Compared with the conventional dental polymers, the fiber-modified and the high impact polymers are successful in their application because of their high elastic modulus and flexural strengths. Most of the modified specimens showed better flexural strength than that of the conventional acrylic resin. Specimens of the high impact resin showed the highest flexural strength, followed by the fiber-modified and then the TiO_2- modified specimens. These results were discussed by another study which stated that, fiber reinforcements reinforce denture base polymer more than non-impregnated fiber reinforcements [1,29]. The transverse strength of the heat-cured denture base resins was improved by using metal wire and glass fibers as reinforcing agents. However, the addition of unidirectional glass fibers was significantly more effective method to increase the transverse strength of denture base resins [18,28,30-33].

A study found that there was a reduction in the transverse strength of fiber-reinforced acrylic resin. The authors concluded that, this reduction in the transverse strength of the test specimens is caused by other factors, for example, by improper impregnation of PMMA into the fiber, rather than by inadequate adhesion [34]. The unidirectional glass-fiber reinforced composite had a reinforcing effect on the flexural strength at the proportional limit of the denture base resin [35]. The random orientation of the fibers is technically easier and can be followed in the dental laboratory routinely [11].

The flexural modulus of the test specimens depends on the type of reinforcement. This can be explained by the stiffness of the fibers exceeds that of the homogenous polymer matrix. So the results of the flexural modulus recorded for the fiber-modified and the high impact acrylic resins were indicative of the reinforcement of the corresponding additives. This can be explained by the capability of the reinforcing fibers to withstand the compressive moduli of reinforcing fibers compared with the conventional acrylic resins [13].

The acrylic resins reinforced with rubbery inclusion is preferred to unreinforced conventional acrylic resins, since its formulation has properties comparable with those of the best proprietary materials, and it meets the requirements of impact strength with minimal decrease in Young modulus [12]. The high impact and the fiber- modified samples showed the highest values of elastic modulus followed by the conventional and TiO_2-modified acrylic resin samples. Because the modulus of elasticity is the ratio of the stress to the strain for a given stress, the greater the value of the modulus, the stiffer the material will be, and the considerable stress must be induced before

a notable strain or deformation results [18]. It was reported that fiber reinforcement yielded higher elastic modulus for reinforced heat-polymerized acrylics [36]. Also, some researchers concluded that glass fibers reinforcement was greatly reducing the modulus of elasticity of the acrylic resin [30]. This conflict in these results may be related to the time of fiber impregnation where prepolymerized fibers improved the overall mechanical properties of reinforced auto polymerized acrylic resins more than post polymerized fibers [36]. The reduction in the modulus of elasticity may be a reflection of the type of reinforcement, for example the incorporation of the TiO_2 nanoparticles.

Fracture toughness test was conducted because one of the common causes of denture fracture is continued flexing of the base during function, which leads to crack development. The fracture stems from the initiation and propagation of a crack, and it require the presence of a stress raiser or localized stress [37]. In this study, the presence of TiO_2- modification would produce a decrease in the toughness values; this might be due to the lack of adhesion between the metal particles and the matrix, which might cause unwanted dispersion of cracks around the metal particles. Specific areas of the prostheses, such as a fixed partial denture connectors and the palatal areas of dentures may be custom designed to yield enhanced properties of elastic modulus and toughness, making the prostheses more resistant to failure [23]. There was a high positive correlation between flexural strength, flexural modulus and fracture toughness. There was a negative correlation between deformation and flexural modulus. This indicated that the materials behave as a brittle fracture. The property of deflection critically affected the other properties of the acrylic resin materials. Even when fracture force increases, a decrease in deflection will result in reduced toughness.

Conclusions

Within the limitations of this laboratory testing conditions of this study, the following conclusions were drawn:

1. The reinforcing materials did not affect monomer releasing.

2. The deflection rates were comparable for all acrylic resin materials.

3. The flexural strength of the acrylic resin was improved after modification with glass fibers.

4. The flexural modulus didn't change significantly in all groups.

5. The toughness of the acrylic resin could be improved by addition of glass fibers.

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