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The residual monomer content and mechanical properties of CAD\CAM resins used in the fabrication of complete dentures as compared to heat cured resins

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Abstract

Background: The utilization of computer-assisted designing and computer-assisted milling CAD\CAM resins in the fabrication of removable prostheses is a modern-day concept that offers many advantages over the use of the traditional polymethylmethacrylate (PMMA).

Aim: This study instigated some of the mechanical properties of CAD\CAM denture base resin including the amount of residual monomer.

Methods: This study was conducted at the Faculty of Dentistry, King Abdulaziz University from October 2016 to February 2017. A total of seventy rectangular specimens were fabricated (group A: 35 heat-cured PMMA and group B: 35 CAD/CAM pre-polymerized acrylic resin blocks). The flexural strength and surface hardness were tested while the residual monomer content at baseline, two-day and seven-day intervals was estimated using gas chromatography (GC). Means and standard deviations were determined for each group as well as independent-samples t-test and ANOVA with repeated measures for comparison between the groups and subgroups of varying time intervals.

Results: Heat cured PMMA (A), displayed higher flexural strength and low value flexural modulus compared to CAD/CAM acrylic resin denture base material (B). Student t-test indicated highly significant differences ($p<0.001$) of the flexural strength ($t=37.911$) and flexural modulus ($t=88.559$). The surface hardness of group (B) was significantly higher compared to group (A) as indicated by the t-test ($t=20.430$). Higher release of the monomer content was detected by GC in group (A) at different time intervals with a statistically significant difference ($p<0.001$) in residual monomer content.

Conclusion: CAD/CAM resin may be considered suitable for use in the construction of denture bases.

Keywords: Acrylic resin, Dentures, Residual monomer, Flexural strength, Surface hardness

1. Introduction

Heat cured polymethylmethacrylate (PMMA) resin is the most widely used material for the construction of removable prostheses (1) due to its superior physical and chemical characteristics, ease of processing and reasonable cost. However there are a few limiting properties, most importantly is the release of residual methyl methacrylate monomer (MMA) which affects the dimensional stability, the adherence of oral bacteria such as Candida albicans to the resin and the prolonged chair side appointments required for the fabrication of the prostheses (2). Such limitations have led general practitioners to shy away from treating edentulous patients (3-5). The presence of residual monomer in denture base resins adversely affects both the mechanical properties and the biocompatibility of these resins as leaching out of the monomer gives rise to allergic reactions with symptoms such as burning sensations, stomatitis, oedema and ulceration of the oral mucosa (6). The employment of computer-assisted designing and computer-assisted milling (CAD\CAM) technology in the fabrication of removable prostheses may eliminate such disadvantages (7). The application of CAD\CAM technology is increasing in dental laboratories and

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clinical practices alike (8, 9). CAD\CAM systems offer numerous clinical benefits since the PMMA pucks used for the milling of denture are polymerised under high temperature and pressure, a process that promotes the formation of longer polymer chains leading to a higher degree of monomer conversion and lower values of residual monomer as well as minimal porosity (10-16). The level of residual MMA in denture base resins has been investigated through various methods such as infrared spectroscopy (IR), high performance liquid chromatography (HPLC) and gas chromatography (GC) (17-19). However, some limitations are inherent to some tests. For example, Infrared spectrophotometry suffers from the overlapping of absorption peaks in the complex spectra and ample preparation for HPLC analysis is complex. On the other hand, GC is a convenient test as it separates the monomer without decomposition, requires short run times and offers a high sensitivity range (20). This study aimed to evaluate some of the flexural strength and hardness properties of CAD\CAM denture base resin and to determine the amount of residual monomer content in these resins using the gas chromatography analytical technique GC.

2. Material and Methods

2.1. Study design

This study was carried out from October 2016 to February 2017 at the Faculty of Dentistry, King Abdulaziz University (Saudi Arabia). Resin specimens measuring 65 mm in length, 10 mm in width and 3 mm in thickness were fabricated and consisted of two types: group A (heat cured PMMA) and group B (CAD\CAM pre-polymerized acrylic resin). Group A: The lost wax technique was employed to construct the moulds that were then packed using heat-cured acrylic resin (Vertex RS. Dentimex, Netherlands) according to the manufacturer's instructions then processed in a metal flask. These specimens were processed for 9 hours in a water bath, and kept at a constant temperature of 165 °F (73.5 °C). Conventional cutters and trimmers were used for finishing and polishing. Group B: The CAD/CAM pre-polymerized acrylic resin blocks (Polident d.o.o. Volčja Draga 42, Sl-5293 Volčja Draga, Slovenia) were cut with an Isomet saw (Isomet, Buehler, USA) under running water then ground on 800- grit SIC. All specimens were stored in distilled water at 37 °C for 24 hours.

2.2. Mechanical and chemical testing

Specimens were divided into three subgroups: Subgroup 1: Flexural strength and flexural modulus test (10 specimens); Subgroup 2: Surface Hardness test (10 specimens); Subgroup 3: Gas chromatography GC analysis (15 specimens).

2.3. Flexural strength test

Flexural strength testing was conducted using the Instron testing machine (Instron 5944, 2kn. 825 University Ave, Norwood, MA, USA). This was through applying the three-point testing design with a centrally loaded specimen beam at a cross-head speed of five mm/min over a two-point support span that was set at thirty mm. The force required for break samples was recorded using the universal testing machine software (Bluehill3, V3.12, Illinois Tool Works Inc. USA). The flexural strength was calculated applying the following equation (1):

$S= 3.P.L/ 2.b.d^2$

Where:
S: flexural strength (MPa)
P: maximum load applied (N)
L: support span length (mm)
b: width (mm)
d: thickness (mm)

The flexural modulus (E) was computed using the equation (2):

$E = FL^3/4ybd^3$

Where:
F: load applied
Y: deflection
L: support span length (mm)
b: width (mm)
d: thickness (mm)

2.4. Surface Hardness Test

Surface hardness was determined by means of a digital Micromet hardness tester (Micromet 6040, Buehler LTD, Illinois 60044. USA) with load of 50 gf for 10 seconds (dwell time) applied using a blunt pointed indenter.

Measurements were obtained and calculated using the built-in software (Buehler OmniMet MHT 7.3, Buehler LTD, Illinois 60044. USA). Five areas per sample were tested and the mean value of these readings was measured to determine hardness.

2.5. Gas chromatography (GC)

Gas chromatography was used to measure the residual monomer content in all specimens immediately following processing (subgroup a), at 2 days and 7 days of storing in distilled water (subgroup b and c respectively). The solvents of analytical reagent grade (Merck, Frankfurter Str. 250, 64293 Darmstadt, Germany) were used. A solution of hydroquinone (HQ), methanol and acetone was prepared with a concentration of 20 ppm as a stabilizer of MMA. Internal standard (I.S.) solution was prepared using 750 mg butanol and methanol to a total volume of 25 ml. The concentration of the prepared I.S. in the solution was approximately (30mg/ml). Working standard solution consisted of 142 mg of MMA and methanol with a concentration of (14.2 mg/ml). 100 µl of working standard solution, 200 µl of the internal standard solution were added to 10ml methanolic solution to give a final concentration of 0.142 mg/ml.

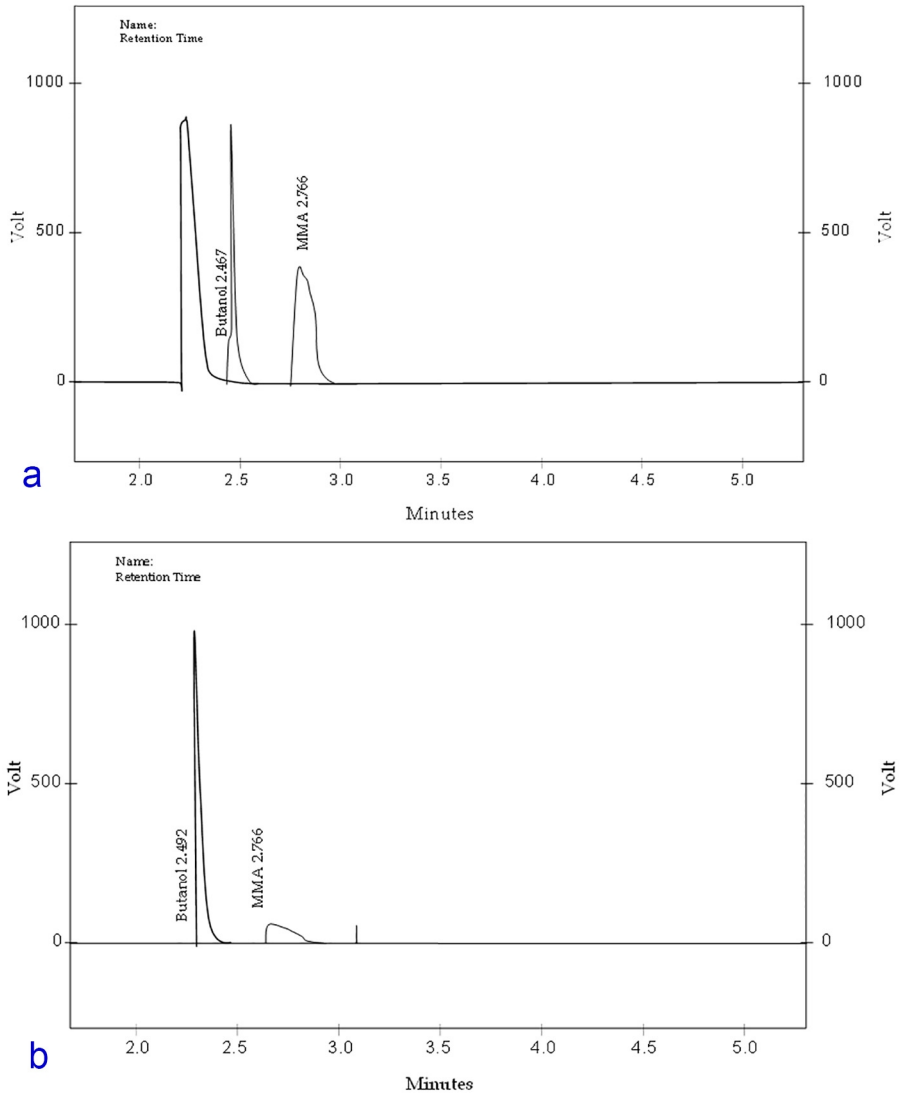

Figure 1. Peak of butanol (internal standard) and retention time of MMA by gas chromatography of group (A) and group (B)

2.6. GC sample preparation

six hundred fifty mg of resin (groups A and B) were placed into the acetone diluting solution and stirred with stirring bars for 72 hours at room temperature to create slurry. Two ml of this slurry was added to the final methanol diluted solution and five ml of this mixture slurry was transferred by pipette to a glass centrifugation tube. The slurry was centrifuged (3000 rpm for 15 min) then one µl was tested using the GC (Shimadzu, Chiyoda-ku, Tokyo 101, Japan). This system utilized a gas chromatograph with a flame ionization detector (FID) on a fused-silica capillary column RTX-5 (30 m × 0.23 mm i.d. 0.25 µm ft.) with 5% phenylmethylpolysiloxane coating and split mode (10:1) injection at 200 °C, the column temperature. The temperature was raised from 40 °C to 100 °C at a rate of 20 °C/min. The carrier gas (N2) was set at a flow rate of 1.2 ml/min and detection at 250 °C with a ratio of H2/air at 45/450. The standard sample peak retention time and area were compared to the test sample to calculate the concentration (Figure 1). The content of residual monomer was determined via standard calibration curve. The monomer content in the sample solution and in the test specimens (groups A and B and their subgroups) was calculated (3).

2.7. Statistical analysis

In this comparative study, means and standard deviations were determined for each group as well as independent-samples t- test and ANOVA with repeated measures for comparison between the groups and subgroups of varying time intervals.

3. Results

The mean distribution and standard deviation values of flexural strength, flexural modulus and surface hardness are presented in (Table 1). The mean value of flexural strength in group (A) was 62.38 ± 1.73 MPa, which was significantly higher than that of group (B) 34.05±2.32 MPa as indicated by t-test (t=37.911) ($p<0.001$). Student t-test also calculated the mean value of flexural modulus at 5% level of group (A) to be significantly lower than group (B) (t=88.559) ($p<0.001$). Results are displayed in Figure 2. The surface hardness of group (B) revealed a considerably higher mean value at 22.41±1.50 compared to group (A) at 13.22±0.88, with a statistical significant difference at 5% level among the two groups according to the t-test (t=20.430) ($p<0.001$). Results are displayed in Figure 3. Table 2 presents the GC results of the mean residual monomer content released at different time intervals in all samples (groups A, B and their subgroups). The mean value of residual monomer content in group (A) was higher than those of group (B). ANOVA, with repeated measures was used for comparison within the subgroups at different time intervals and revealed a statistically significant difference (F=174.194 and 492.759) ($p<0.001$). The student t-test displayed a high statistically significant difference ($p<0.001$) at 5% level as shown in Table 2.

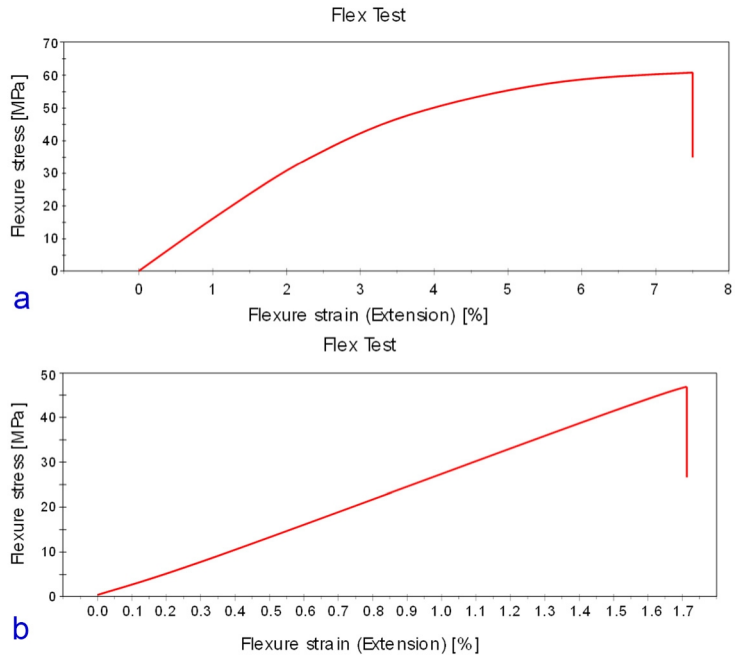

Figure 2. Flexure strength of group (A) and group (B).

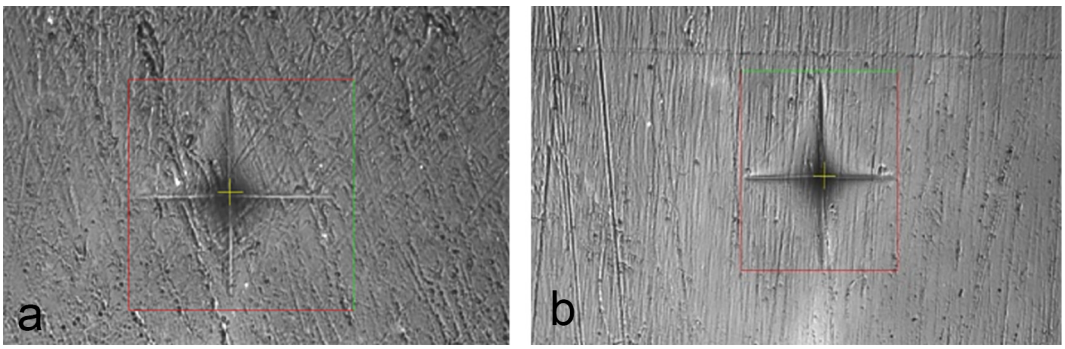

Figure 3. Surface hardness of group (A) and group (B).

Table 1. Mean value, standard deviation and t-test for flexural strength, flexural modulus and surface hardness of heat cured and CAD/CAM acrylic resin denture bases

Resin	Flexural strength Mean ± SD (MPa)	flexural modulus Mean ± SD (MPa)	Surface Hardness Mean ± SD (MPa)
Heat Cure PMMA	62.38 ± 1.73	1.55 ± 0.06	13.22 ± 0.88
CAD/CAM resin	34.05 ± 2.32	2.85 ± 0.01	22.41 ± 1.50
t - test	37.911*	88.559*	20.430*
p-value	<0.001*	<0.001*	<0.001*

*Statistically significant at $p \leq 0.05$

Table 2. The mean value, standard deviation of residual monomer content of group A and group B in gas chromatography at different time intervals, t and p values for Student t-test for comparing between the two groups. F test (ANOVA) with repeated measures for comparison between different periods.

Resin	Gas chromatography			F	p-value
	Baseline	2 Days	7 Days		
Heat Cure PMMA	17.10±0.38	16.04±0.13	13.54±0.46	174.194*	<0.001*
CAD/CAM resin	1.61±0.05	1.03±0.06	0.90±0.01	492.759*	<0.001*
t	90.261*	235.221*	61.934*		
p-value	<0.001*	<0.001*	<0.001*		

*Statistically significant at $p \leq 0.05$

4. Discussion

CAD/CAM technology has multiple prosthetic applications including the fabrication of intra-coronal and extra-coronal restorations, crowns, fixed partial dentures and more recently, the construction of complete denture and maxillofacial prostheses (21). CAD/CAM allows the use of novel materials with improved characteristics (9) but it is crucial to understand and assess the mechanical properties of these newer materials. CAD/CAM denture base acrylic resin is supplied as pre-polymerized blocks which are produced in industrially controlled conditions with standardized pressure and temperature parameters. These blocks, which are easily milled, have gained popularity in recent years in the fabrication of removable prostheses. It is crucial to examine the mechanical properties such as flexural strength, flexural modulus and surface hardness as these replicate the clinical loading patterns applied to a denture intra orally. This is an important factor to consider as patients use their removable prostheses for extended periods of time (8, 22-24). In the current investigation, the heat cure acrylic denture base of group (A) showed a higher flexural strength with low flexural modulus compared to the tested CAD/CAM acrylic resin denture base material of group (B) despite the similarity in the PMMA composition of both resins. These findings could be due to the higher release of the monomer content, which was detected by GC in group (A), that adversely affects the mechanical properties as a result of plasticizing the polymeric chain, resulting in greater resin deformation under loading (20). The decrease of flexural strength with increase flexural modulus of group (B) is probably a result of the processing method under high temperature – pressure leading to a low residual MMA concentration. It has been reported that these processing conditions decrease the intermolecular distances and reduce the free volume (25). Surface hardness indicates the density of the material and its resistance to wear and/or scratching which reflects on the dental prosthesis during its function and cleaning (14). The results of the present study showed a significant difference in surface hardness of the CAD/CAM resin compared to heat cured PMMA resin. These may be

attributed to the polymerization process of each resin as the heat cure is polymerized by additional (free radical) polymerization and leads to the formation of a partial cross-linked polymer chain which results in the superior hardness. On the other hand, the high temperature and high-pressure conditions for the polymerization of CAD/CAM resins and the addition of inorganic fillers restrict dimensional polymerization shrinkage and enhance the CAD/CAM resins mechanical properties including hardness and wear resistance (24-26). Such properties also decrease surface deterioration and the adhesion of bacterial plaque (14, 27). High monomer content has detrimental effects on the mechanical properties of the resin, reduces the glass transition temperature which renders the resin softer and more flexible and adversely affects the prostheses clinical performance. Determining the content of residual monomer in denture base resin using gas chromatography is a simple and rapid yet reliable technique.

5. Conclusions

Gas chromatography analysis of residual MMA in polymerized denture base material offers sufficient accuracy to measure the residual monomer in acrylic denture base materials with minimal sample preparation. The findings of the current study indicate a reduction of monomer content in CAD/CAM resin which could be attributed to the method of polymerization under high pressure. CAD/CAM acrylic resin denture base is considered as a clinically suitable resin for the construction of denture bases. Further investigation of the mechanical properties of CAD/CAM materials are necessary to assess other aspects prior to clinical trials.

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Conflict of Interest:

There is no conflict of interest to be declared.

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