EFFECT OF VARYING POWDER LIQUID RATIOS AND CURING CONDITIONS ON THE RESIDUAL MONOMER CONCENTRATION OF HEAT CURE ACRYLIC RESIN

[1]SALEHA NISAR
[2]FAISAL MOEEN
[3]YAWAR HAYAT KHAN

ABSTRACT

Objective of the study was to determine the effect of varying powder liquid ratios and curing conditions on the residual monomer concentrations of heat cure acrylic denture resin.

Sixty four disc shaped acrylic resin specimens were processed at four powder-liquid ratios (2.22, 2.00, 1.80 and student-ratio) and cured in a heated water bath using 4 different curing regimes. Curing cycles 1A and 1B initiated curing of specimens at room temperature followed by a terminal boil for 60 and 30 minutes respectively. Cycles 2A and 2B initiated curing at 70°C and 100°C respectively, without any terminal boil. The residual monomer concentrations were determined at intervals of 24, 48 and 72 hours using a UV Spectrophotometer.

Regression analysis indicated a moderately strong negative correlation (-0.515) between time and residual monomer concentration which suggests residual monomer concentration decrease with storage time. Group 4 (student-ratio) and curing by cycle 2B (placing the assembly in the water bath directly at 100°C) showed the highest overall residual monomer concentrations while group 1 (highest powder-liquid ratio) and curing by cycle 1A showed the least overall residual monomer concentrations. Independent t tests between groups however revealed mean residual monomer concentration of samples cured by cycle 2B (0.32±0.38) to be greater than the mean (0.06±0.07) of samples cured by Cycle 1A (p value= 0.02) suggesting that cycle 1A will produce less residual monomers irrespective of the powder-liquid ratio.

It was concluded that students dispensing acrylic powder and liquid monomer without calculations would lead to higher residual monomers if a proper curing regime is not followed. Using a 60 minute terminal boil in the polymerization process is strongly recommended. Storing the acrylic denture bases for at least 24-48 hours in water before use will decrease residual monomers being leached into the oral environment.

INTRODUCTION

Inspite the extensive use of Polymethly-methacrylate (PMMA) in dentistry, complications associated with their inherent property related limitations and processing have been documented.[1] One of the problems associated with acrylic dentures is the presence of residual monomers following fabrication.[2] Studies have shown that polymerization of methylmethacrylate is never quite complete and despite long curing cycles of polymerization minimal amounts of un-polymerized residual monomer can always be found.[3,4] Mechanical and biological consequences of un-polymerized residual monomers have also been studied extensively.[5,6] A number of studies have reported various degrees of in-vitro cytotoxicity and in- vivo allergic responses to leached residual monomers.[7-10] Mechanical properties of acrylic denture base resins, such as hardness and flexural strength, have also been known to be adversely effected by the extent of un-polymerized monomers within the matrix.[11,12]

In the past few years, various processing methods and material variations have been introduced to improve the mechanical, physical and biological properties of cured acrylic resins.[13-17] Researchers have introduced

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[1] Dr Saleha Nisar, BDS, M.Phil (Dental Materials), Lecturer, Department of Dental Materials. Islamic International Dental College, Riphah International University, Islamabad.
Email:salehanisarhaq@gmail.com
Email: saleha.nisar@riphah.edu.pk Cell: +92-321-5017738

[2] **Correspondence:** Dr Faisal Moeen, BDS, MSc (Singapore), Associate Professor, Department of Dental Materials. Islamic International Dental College, Riphah International University, Islamabad. Email: Faisalmoeen_is@yahoo.com
Faisal.moeen@riphah.edu.pk Cell: +92-300-8546021

[3] Dr Yawar Hayat Khan, BDS, MSc (London), MHPE (Maastricht), Associate Professor, Department of Dental Materials. Islamic International Dental College, Riphah International University, Islamabad.

Received for Publication: October 30, 2015
Approved: November 14, 2015

a number of curing techniques to achieve a maximum monomer to polymer conversion to produce acrylic with superior properties.[18] Methods such as the introduction of microwaves and visible light activated curing techniques to reduce the curing time, have revolutionized the science of denture acrylics. However, curing acrylic dentures in the conventional water bath still remains the most widely used method.[19]

According to the ISO 1567 standard, the maximum acceptable quantity of residual monomers in acrylic should not be more than 2.2% by weight.[20,21] However, clinical reports have shown the presence of variable amounts of residual monomers in an acrylic dentures base.[8,22-24] Factors such as temperature, time, initiator concentration, curing environment (water bath v/s microwave), pressure, mixing ratio and denture base thickness have an effect on the residual monomer content.[24,25] One additional factor affecting the cytotoxicity of acrylic is the polymer powder to monomer liquid ratio.[26,27]

The amount of residual monomer in an acrylic resin denture base, has been determined using various methods. Fourier Transformation Infrared (FTIR), Ultraviolet and Visible Spectrophotometry, High Pressure Liquid Chromatography and Gas Chromatography (GC) have been most popular.[27-30] The aim of this study is to determine and compare the effects of varying powder liquid ratios and curing cycles on the residual monomer concentration of cured acrylic resins. Residual monomer concentrations would be determined for acrylic specimens made at the manufactures recommended powder-liquid ratio as well as those dispensed by under-graduate dentistry students.

METHODOLOGY

a) Preparation of a standard mould

Poly-tetra-fluoroethylene (Teflon) rods having a diameter of 38.1mm (1.5 inches) were cut into 12 discs each having a thickness of 25.4mm. These Teflon discs were subjected to a milling process in order to create a 4mm deep hollow cavity with a precise diameter of 32mm. The created cavities would serve as moulds for the fabrication of our heat cured acrylic discs.

b) Preparation of acrylic specimens

The heat cure acrylic powder was mixed with methyl-methacrylate liquid monomer according to ratios mentioned in Table 1. The heat cure liquid was placed into a clean dry mixing vessel and the powder was sprinkled onto the liquid over a period of 30 seconds according to manufacturer recommendations.[31] A spatula was used to mix the material which was finally packed at the doughy stage into the moulds and each covered with a polyethylene sheet. The dental flasks were placed in a hydraulic bench press (Dental Hydraulic Flask Press, BISON, Intensiv Industries, India) at 80 bars of pressure for 25 mins. The flasks were then immersed in water and cured using an electrically controlled water bath according to the regimes mentioned in Table 2.

The four groups (G1-G4) are based on varying powder liquid ratios, with each group having to undergo four curing cycles (1A, 1B, 2A and 2B). Sixteen specimens of acrylic discs were created for each group translating to a total of 64 discs. Group G4 constituted specimens processed at un-calculated powder-liquid ratios dispensed by 16 final year BDS students at the Islamic International Dental College, Islamabad, Pakistan. The amount of monomer leached from each specimen was measured at an interval of 24hrs, 48hrs and 72hrs in the UV spectrophotometer.

c) Curing cycles for the test specimens

- **Cycle 1A:** The flasks were immersed in a water bath at room temperature (25 degree C). The temperature was then gradually increased to 70°C (165°F) and maintained for 90 minutes. The temperature was then raised to 100°C (212°F) and maintained for 60 minutes.
- **Cycle 1B:** The flasks were immersed in a water bath at room temperature. The temperature was gradually increased to 70°C and maintained for 60 minutes. The temperature was then raised to 100°C and maintained for 30 minutes.
- **Cycle 2A:** The flasks were immersed in a water bath having a temperature of 70°C and the temperature was gradually increased to 100°C and maintained for 30 minutes.
- **Cycle 2B:** The flasks were immersed in a water bath having a temperature of 100°C and cured for 30 mins.

Following the completion of the mentioned curing cycles, the flasks were allowed to cool to room temperature. The acrylic specimens were removed and finished using an acrylic trimmer and sandpaper. Polishing of the specimens was done with pumice slurry on a lathe polishing buff.

d) Formation of a standard calibration curve

A stock solution of 3% v/v methylmethacrylate was initially prepared by dissolving 3ml of methylmethacrylate in 100ml of distilled water. From this stock solution, a series of aliquots ranging from 0.5% to 3.0% were created by simply diluting the stock with specific volumes of distilled water. Absorbance of these solutions was determined at 210nm using the UV spectrophotometer to obtain a standard graph of concentration against absorbance (Fig 1, Table 3). The standard calibration curve would then be used to determine the amount of methylmethacrylate leached from the 64 acrylic discs.

e) Residual monomer determination of acrylic specimens

Each of the 64 acrylic specimens was immersed in 15ml of distilled water and subjected to UV spectroscopy at 210nm after intervals of 24, 48 and 72 hours. The amount of residual monomer leached into the distilled water was analyzed and values are compared with the standard graph.

RESULTS

The mean of the residual monomer measurements for the 4 groups are mentioned in table 4 and represented in Fig 3. A linear regression analysis was carried out showing values of residual monomer concentration and variables of powder-liquid ratios, curing cycles and storage time are statistically significant with a p-value of 0.000. Moderately strong negative correlation (-0.515) exists between time and residual monomer concentration, indicating residual monomers decrease with storage time. Group 4 (student-ratio) and Cycle 2B (insertion of the curing assembly in the water bath at 100°C) showed the highest overall residual monomer concentrations, while Group 1 (highest powder-liquid ratio) and Cycle 1A (immersing the flask in water bath at room temperature, gradually increasing the temperature to 70°Cand maintaining it for 90 minutes, followed by increasing the temperature to 100°C and maintaining it for 60 minutes) showed the least overall residual monomer concentration.

TABLE 1: POWDER LIQUID RATIOS

	Powder	Liquid
G1	23.4gm	9ml
G2	23.4gm	10ml
G3	23.4gm	11ml
G4	Students ratio	

TABLE 2: CURING CYCLES OF THE RESINS T0: START TEMPERATURE OF WATER T1AND T2:TEMPERATURE OF WATER DURING POLYMERIZATION CYCLE T0, T1, T2: MAINTAINING TIME AT THE RELATIVE TEMPERATURES

	T0	T0	T1	T1	T2	t2
1A	25°C	0 min	70°C	90 min	100°C	60 min
1B	25°C	0 min	70°C	60 min	100°C	30 min
2A	70°C	0 min	100°C	30 min	—	—
2B	100°C	0 min	100°C	30 min	—	—

TABLE 3: DATA FOR STANDARD GRAPH

Concentration	Absorbance Values				Mean Absorbance
0.5%	0.98	1.02	0.99	1.01	1.00
0.6%	1.11	1.16	1.16	1.13	1.14
0.8%	1.25	1.30	1.26	1.23	1.26
1%	1.46	1.51	1.49	1.54	1.50
1.2%	1.76	1.75	1.77	1.72	1.75
1.4%	2.05	2.02	2.01	2.00	2.02
1.6%	2.33	2.36	2.38	2.37	2.36
1.8%	2.68	2.65	2.68	2.59	2.65
2%	2.84	2.86	3.0	3.30	3.00
2.5%	3.67	3.71	3.81	3.81	3.75

TABLE 4: MEAN RESIDUAL MONOMER CONCENTRATION

No. of Sample		Concentration Values			
Group 1					
Time		Cycle 1A	Cycle 1B	Cycle 2A	Cycle 2B
24 hr	4	0	0.062	0.274	0.305
48 hr	4	0	0.051	0.016	0.070
72 hr	4	0.040	0.086	0.038	0.044
Group 2					
Time		Cycle 1A	Cycle 1B	Cycle 2A	Cycle 2B
24 hr	4	0.029	0.102	0.33	0.545
48 hr	4	0.016	0.109	0.04	0.126
72 hr	4	0.02	0.023	0.046	0.024
Group 3					
Time		Cycle 1A	Cycle 1B	Cycle 2A	Cycle 2B
24 hr	4	0.058	0.197	0.244	0.661
48 hr	4	0.02	0.03	0.023	0.155
72 hr	4	0.009	0.023	0.064	0.068
Group 4					
Time		Cycle 1A	Cycle 1B	Cycle 2A	Cycle 2B
24 hr	4	0.197	0.520	1.010	1.062
48 hr	4	0.042	0.120	0.040	0.52
72 hr	4	0.027	0.176	0.073	0.123

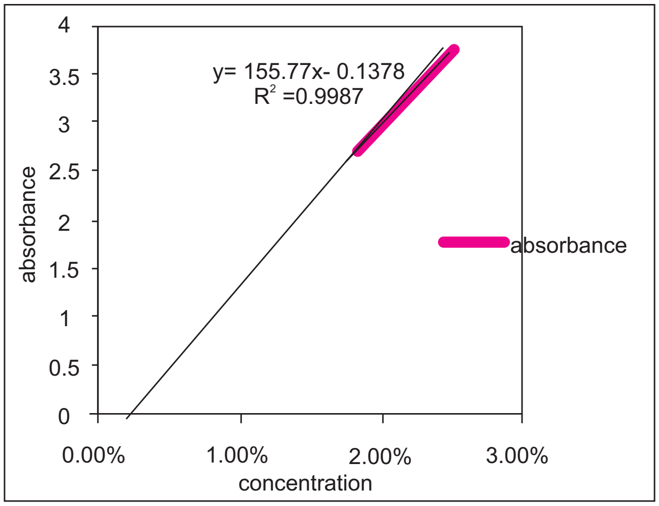

Fig 1: Standard Calibration Curve

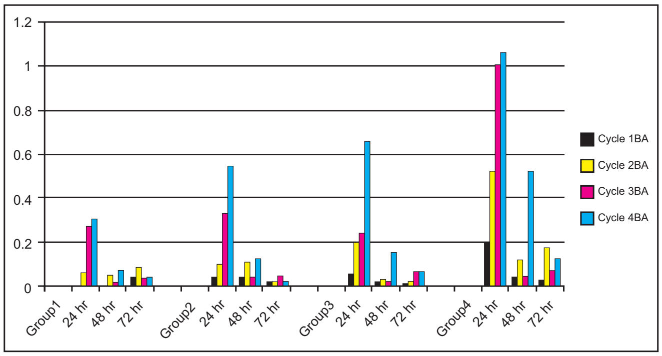

Fig 2: Graphical representation of mean residual monomer concentration of the four groups

Independent sample t tests were used to compare the residual monomer concentration of samples cured using Cycle 1A to those cured with Cycle 2B, as well as Cycles 1A and 1B. The mean residual monomer concentration of samples cured by Cycle 2B (0.27±0.313) was found to be greater than the mean residual monomer concentration (0.04±0.05) of samples cured by Cycle 1A (p value= 0.00). Based on these results it may be suggested that Cycle 1A will produce less residual monomers, irrespective of the powder liquid ratio, compared to Cycle 2B. Hence the powder liquid ratio used by the students (Group 4) would not influence the residual monomer concentration if an appropriate curing cycle (such as Cycle 1A) is adhered to.

The mean residual monomer concentration of samples cured by Cycle 1A (0.04±0.05) was found to be less than the mean residual monomer concentration (0.125±0.132) of samples cured by Cycle 1B (p value 0.00). These results suggest that decreasing the curing time and the terminal boil period by 30 minutes produces more residual monomers.

DISCUSSION

In this study UV spectrophotometer was used to determine the amount of monomer released into distilled water.[22,32] The results suggest that curing cycles have a higher positive correlation (0.361) as compared to powder-liquid ratio (0.30), which is in agreement with Jerolimov et al[33], who stated that the choice of curing cycle has much greater influence on the level of residual monomer as compared to the mixing ratio.

Group 4 of our study constituted samples prepared by 4th year undergraduate students dispensing and mixing the acrylic powder and liquid under supervision. It was noted that the students mixed the acrylic resin normally without following any ratio or measuring system. A powder-liquid ratio of 2.34:1 is recommended by the manufacturer, which limits the volumetric contraction during polymerization as well as excess un-polymerized monomer.[2] When an acrylic denture is prepared with a higher proportion of polymer at a higher powder-liquid ratio, the levels of residual monomer appears to be low.[34] As shown in Table 4 and Fig 2, decreasing the powder-liquid ratio from group 1 to group 3, caused an increase in residual monomer concentrations. The lowest values of residual monomer were found in Group 1 where the powder-liquid ratio was highest. The highest residual monomer concentration was seen in the student group which indicated students seem to dispense more liquid monomer resulting in a lower powder-liquid ratio.

As the use acrylic resins has dramatically increased, different curing cycle have been introduced with the express purpose of achieving a higher monomer to polymer conversion. Variations in curing temperatures and soak times have been documented. However, in this study 4 short cycles were selected based on the cycles commonly used by students and laboratory technicians.

In Cycle 1A and 1B the flasks were kept in a water bath at room temperature and the temperature was gradually increased. The rate at which the material reaches its maximal temperature has to be carefully controlled in order to avoid gaseous porosity.[35] It seems from our results that gradually increasing the temperature and curing time lead to a decrease in the residual monomer content, whereas, placing the flasks directly at high temperatures caused an increase in residual monomer content which is also in agreement with other studies.[36]

Although the powder-liquid ratio used in group 2 was recommended by manufacturer, and the residual monomer content in group 2 increased from cycle 1A (0%) to cycle 2B (0.305%) in 24 hours. These results indicate that short curing cycles and curing acrylic resins at increased temperatures release more residual monomers. The initiator, benzoyl peroxide decomposes above 60°C, giving free radicals which start and propagate polymerization.[37] The decomposition of benzoyl peroxide is temperature dependent, hence in long curing cycle greater decomposition of benzoyl peroxide results in greater number of polymer chains. Conversion of monomer to polymer is time dependent and the rate of conversion is greatly increased by increasing the temp from 70°C to 100°C.

According to Harrison and Huget, the most effective polymerization cycle with minimal levels of residual monomer was a 7 hour incubation in water at 70°C followed by a post polymerization treatment at 1h at 100°C.[38] Although many authors prefer long curing cycles, Bayraktar et al[39] found that long curing cycles (9 hours at 70°C) without post polymerization terminal boil showed higher content of residual monomer as compared to a shorter cycle followed by a short terminal boil (20 minutes at 70°C, followed by 22 minutes at 100°C). Hence to reduce the final residual monomer content, a final post polymerization treatment (terminal boil, microwaving for few minutes or immersing in water for at least 24 hours) is most effective.[8,39-41] Cycles 1A and 1B underwent a terminal boil post polymerization treatment, which could be the reason for low residual monomer content.

As the polymerization reaction proceeds, the amount of monomer and free radicals decrease and due to increase in viscosity and degree of polymerization, it becomes difficult to bring the monomer and free radical together. Hence the diffusion of monomer becomes the rate–limiting step. Increasing the temperature causes the molecules to move faster and the polymerization reaction will be more complete.

The release of MMA into water decreases with time. In this study maximum release of residual monomer occurs in the first 24 hours. The decrease of release of MMA in water could be due to hydrolysis of methylmethacrylate to methacrylic acid and methanol as well as due to continued polymerization of the resin.[42]

The highest amount of residual monomer content was leached from Group 4. It seems that our under-graduate students dispense more liquid monomer than required. If such a mix is to produce a processed denture base with minimal amounts of residual monomers, a water bath curing regime involving a gradual increase in temperature and curing time followed by a terminal boil for 60 minutes should be adopted. Ideally students should follow powder-liquid ratios recommended by manufacturers and hence adequate gauging facilities should be made available at dental colleges.

CONCLUSIONS

1. Students should follow appropriate dispensing techniques at the recommended powder liquid ratios.
2. Curing regimes influence residual monomer concentration more as compared to the powder liquid ratio.
3. Specimens cured using curing cycles starting at room temperature, followed by gradually increasing the temperature seem to exhibit lower values of residual monomers at compared to placing the flasks directly into boiling water.
4. The release of residual monomer decreases with time, hence storing the acrylic resin denture in water for at least 24-48 hours before insertion is recommended.

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CONTRIBUTION BY AUTHORS

1 **Saleha Nisar:**	Study, designed & helped in article writing.
2 **Faisal Moeen:**	Conceived the idea & helped in writing.
3 **Yawar Hayat Khan:**	Helped in planning & reviewing.