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Effect of Varying Curing Regimes and Powder-liquid Ratios on the Flexural Strength and Surface Porosities of Heat Cure Acrylic: An In-vitro Experiment

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Abstract Objectives: To evaluate and compare the effect of varying curing regimes and powder-liquid ratios on flexural strength and surface porosities in heat cure acrylic denture base resin. **Methodology:** Heat cured acrylic specimens (18 x 10 x 3mm) were made according to four powder-liquid ratio groups(2.22, 2.00, 1.80 and student-ratio) and polymerized according to four different curing cycles. Each group consisted of 16 samples with a total of 64 acrylic discs. 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. All discs were tested for flexural strength by the 'short beam' testing method after immersion in water at 37°C for 28 days. Perimeter of each surfacepore was outlined and area of each pore was measured by a SEM. Total area of surface pores was calculated and expressed in percentage form. **Results:** Regression analysis indicated a very weak negative correlation (-0.085) between the powder-liquid ratios and the flexural strength values (p =0.252), indicating that variations in powder-liquid ratio does not affect the flexural strength of the acrylic specimens. Placing the curing assembly directly in the water bath at 100°C for 30 minutes showed the lowest over-all flexural strength and highest porosity percent, while immersing the flask in water bath at room temperature, gradually increasing the temperature from 70°C to 100°C and maintaining it for 60 minutes displayed the lowest over-all percent porosity and highest flexural strength values. A weak positive correlation (0.286) between the groups and percent porosity values (p=0.025) indicate a poor effect of powder-liquid ratio on porosities. **Conclusions:** Flexural strength and percent porosity of acrylic resins are affected more by changes in curing regimes rather than variations in powder-liquid ratio. Increasing the terminal boil period by 30 minutes had a significant effect on reducing percent porosity however did not enhance flexural strength of heat cure acrylic specimens.

Keywords: *Polymethyl-methacrylate (PMMA), polymerization method, flexural strength, porosity, terminal boil*

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1. Introduction

Fractures of PMMA based dentures as a consequence of overloading, impact or cyclic fatigue have been related to design errors as well as the inherent property related limitations of the polymer material [1]. Studies have shown that processing variables such as curing environment, mixing ratio, temperature and time of curing regimes and post processing water storage can all have effects on the flexural strength of denture bases [2]. Incompletely polymerized acrylic resins have been shown to have lower mechanical properties as compared to products been subjected to through polymerization [3,4]. Thus flexural strength measurements can give an assessment of the quality of polymerization and aid in determining the resistance of denture bases to excessive forces [4].

Additionally, factors such as air entrapment during mixing, insufficient mixing, monomer contraction during polymerization, processing temperature higher than 74°C, monomer evaporation during the exothermic curing reaction and insufficient flask compression can cause the formation pores of different shapes and sizes [5,6]. Porosities situated within the matrix of the material can not only weaken the denture base, create high internal stresses but also serve as sites of crack nucleation and crack propagation pathways. Depending on the size and site, porosities can also affect the esthetics of the denture base [7].

In the past few years, various methods and material advancements have attempted to improve the mechanical properties of cured acrylic resins. Researchers have

introduced innovative processing techniques to achieve enhancements in properties of acrylic resins [8]. Methods ranging from introduction of glass fibers to improve mechanical properties, to the use of microwave and visible light activated curing to reduce the curing time have shown favorable results [9,10,11]. However, curing acrylic dentures in the conventional water bath still remains the most widely used method [12].

It has been observed that dental students tend to fabricate acrylic dentures without following the manufacturer's recommended powder-liquid ratios. Material dispensation without proper calculations has been known to elevate residual monomer concentrations and negatively influence mechanical properties of the resin [13]. Additionally, the selection of an optimum curing regime would lead to a maximum monomer to polymer conversion, chain branching and cross linking, consequently leading to a higher average molecular weight of the polymer with better mechanical properties. The objective of this study is therefore to identify an optimum curing cycle and powder-liquid ratio which can lead to a highest flexural strength of the heat-cured acrylic material used at the Islamic International Dental College, Islamabad, Pakistan. Additionally, the effect of curing regimes and powder-liquid ratios on the surface porosities of cured acrylic resins would also be determined.

2. Methodology

2.1. Preparation of Acrylic Specimens

The heat cure acrylic powder was mixed with methyl-methacrylate liquid monomer according to ratios mentioned in Table 1. Following mixing, the material was packed at the doughy stage into a standard mould invested in gypsum. The dental flasks were then placed in a hydraulic bench press (Dental Hydraulic Flask Press, BISON, IntensivIndustries, India) for 25 minutes at 80 bars of pressure.

Table 1. Division of groups according to powder-liquid ratio

	Powder liquid ratios	
	Powder	Liquid
G1	23.4gm	9ml
G2	23.4gm	10ml
G3	23.4gm	11ml
G4	Students Ratio	

Table 2. Curing cycles that the specimens are subjected to

Curing cycles of the resins
T0: start temperature of water
T1 and 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	-	-

The flasks were then immersed in water (covered by 7cm of water) and cured using an electrically controlled water bath according to the regimes mentioned in Table 2.

The four groups (G1-G4) mentioned in Table 1 are based on variations in powder-liquid ratios, with each group having to undergo four curing cycles (1A, 1B, 2A and 2B). 10 acrylic discs were created for each group with a total sample size of 40 discs.

Acrylic discs in Group 2 were processed at the manufacturer's recommended powder-liquid ratio whereas in Groups 1 and 3 the monomer content was kept 10% lower and 10% higher respectively than the manufacturer's recommendation. The students of final year BDS students enrolled at the Islamic International Dental College, Islamabad, Pakistan, were asked to mix acrylic powder and liquid monomer for making specimen in group G4. It was noted that students did not use any measuring system nor did they follow any specific ratios, hence group G4 consisted of specimen that were processed at an un- calculated powder-liquid ratio.

Figure 1. Preparation of the acrylic specimen

2.2. Curing Cycles for the Test Specimens

Cycle 1A: The flasks were immersed in a water bath at room temperature (25°C). The temperature was then gradually increased to 70°C (165°F) over a period of 45 minutes and maintained for 90 minutes. The temperature was then raised to 100°C (212°F) over a period of 30 minutes 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 in 45 minutes and maintained for 60 minutes. The temperature was then raised to 100°C over a period of 30 minutes and maintained at 100°C 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 in 30 minutes and maintained for 30 minutes.

Cycle 2B: The flasks were immersed in a water bath having a temperature of 100°C and cured for 30mins.

Following the completion of the mentioned curing cycles, the flasks were allowed to cool to room temperature. The acrylic specimens were removed from the flasks and finished using an acrylic trimmer and sandpaper. Polishing of the specimens was done with pumice slurry on a lathe polishing buff. The specimens were stored in water at 37°C for a period of 28 days.

2.3. Porosity Analysis

The acrylic disks were viewed under a scanning electron microscope (JEOL JSM-6490A, USA) for the evaluation of porosities. Each specimen was sputter coated with a thin gold coat and surface porosities for each specimen were evaluated at four different fields by the scanning electron microscope at an accelerating voltage of 10KV. The perimeter of each surface pore was outlined (Figure 3) and area of each pore was measured. Total area of surface pores was calculated and expressed in percentage form.

Figure 2. Testing for flexural strength of acrylic specimens

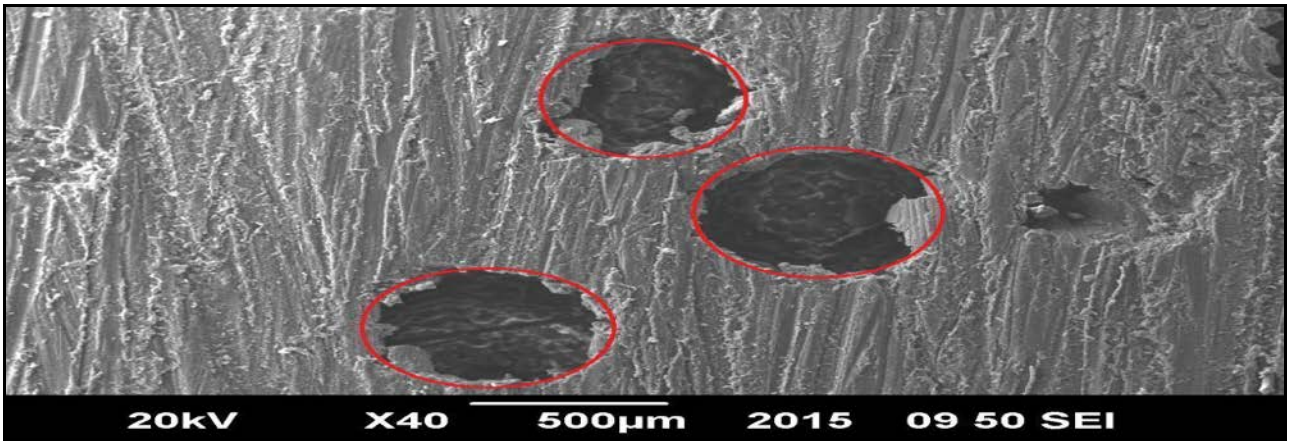

Figure 3. Porosity perimeter outlined for measurement

2.4. Flexural Strength Test

All specimens were subjected to flexural strength testing in a servo-hydraulic universal testing machine (AG-Xplus Series, Shimadzu, Japan) using 3-point loading according toASTM-STP 497 [14].The distance between the specimen supports was 15 mm and the loading force was applied to the specimens at a crosshead speed of 1.5 mm/min until the specimens fractured. Flexural strength (MPa) was calculated using the equation: FS = 3 WL/2 bd2 , where FS is the flexural strength, W is the maximum load before fracture (N), L is the distance between the supports (15 mm), b is the width of the specimen (10mm) and d is the thickness of the specimen (3mm).

3. Results

Mean flexural strength and porosity percent values with standard deviations for the four groups are mentioned in Table 3 and Table 4 and represented graphically in Figure 4 &Figure 5 respectively. Figure 6 & Figure 7 show SEM surface images of specimens cured by the four cycles. A linear regression analysis was carried out showing a statistically significant difference (p=0.001) between curing cycles and powder-liquid ratios with respect to the mean flexural strength and percent porosity. A moderately negative correlation (-0.394) between flexural strength and curing cycles, indicated a decrease in the flexural strength as we move from cycle 1A to cycle 2B. A very weak negative correlation (-0.085) exists between the ratios and the flexural strength values (p =0.252), indicating that variation in powder-liquid ratio does not affect the flexural strength of the acrylic specimens.

Table 3. Flexural strength values of the four groups

Flexural strength values (MPa)							
Groups	**No. of Sample**	**Flexural strength values (MPa)**				**Mean**	**S.D**
Cycle 1A							
Group1	4	53.63	52.42	55.75	52.33	53.53	1.593
Group2	4	52.55	56.31	54.08	49.23	53.04	2.974
Group3	4	53.04	53.8	49.19	57.36	53.35	3.51
Group4	4	57.0	53.31	52.33	46.88	52.38	4.182
Cycle 1B							
Group1	4	52.56	52.97	53.05	52.8	52.85	0.217
Group2	4	50.62	55.77	49.34	57.67	53.35	4.002
Group3	4	69.25	48.02	49	43.73	52.50	11.399
Group4	4	50.61	47.52	56.51	52.75	51.85	3.778
Cycle2A							
Group1	4	53.04	58.25	49.25	37.463	49.50	8.833
Group2	4	47.64	49.51	50.86	48.23	49.06	1.432
Group3	4	42.25	49.19	50.46	48.75	47.66	3.680
Group4	4	53.35	48.17	48.85	45.46	48.96	3.274
Cycle 2B							
Group1	4	47.59	51.62	50.52	47.04	49.19	2.225
Group2	4	49.2	53.05	43.82	45.93	48.00	4.029
Group3	4	41.58	49.2	49.45	53.37	48.40	4.931
Group4	4	30.4	49.25	55.22	53.35	47.06	11.380

Table 4. Porosity percent values of the four groups

Porosity Percent Values							
Groups	**No. of Sample**	**Percent Porosity**				**Mean**	**S.D.**
Cycle 1A							
Group1	4	0.40	0.27	0.35	0.34	0.34	0,07
Group2	4	1.26	0.82	0.38	0.82	0.82	0.44
Group3	4	1.22	1.39	1.39	1.32	1.33	0.09
Group4	4	1.38	1.89	1.56	1.61	1.61	0.25
Cycle 1B							
Group1	4	1.89	1.67	2.2	1.92	1.92	0.26
Group2	4	1.74	2.45	2.08	2.05	2.08	0.35
Group3	4	3.20	2.95	1.89	2.20	2.56	0.65
Group4	4	2.02	2.93	3.06	2.67	2.67	0.56
Cycle2A							
Group1	4	2.70	2.2	3.0	2.90	2.70	0.43
Group2	4	3.21	2.98	3.05	2.68	2.98	0.27
Group3	4	3.91	3.59	3.80	3.90	3.80	0.18
Group4	4	4.25	4.06	4.37	4.32	4.25	0.16
Cycle 2B							
Group1	4	4.24	4.89	3.82	4.01`	4.24	0.57
Group2	4	4.92	5.66	6.54	5.52	5.66	0.81
Group3	4	6.42	5.96	7.22	6.08	6.42	5.66
Group4	4	9.39	6.83	4.22	6.89	6.83	2.5

A strong positive correlation (0.873) between the curing cycles and percent porosity (p=0.000)suggests that porosities increase from cycle 1A to cycle 2B, while a weak positive correlation (0.286) between the groups and percent porosity values (p=0.025) indicate a poor effect of powder-liquid ratio on porosities.

Cycle 2B (placing the curing assembly directly in the water bath at 100°C for 30 minutes) showed the lowest over-all flexural strength and highest porosity percent, while Cycle 1A (immersing the flask in water bath at room temperature, gradually increasing the temperature to 70°C and maintaining it for 90 minutes, followed by increasing the temperature to 100°C and maintaining it for 60 minutes) displayed lowestover-all percent porosity and flexural strength values.

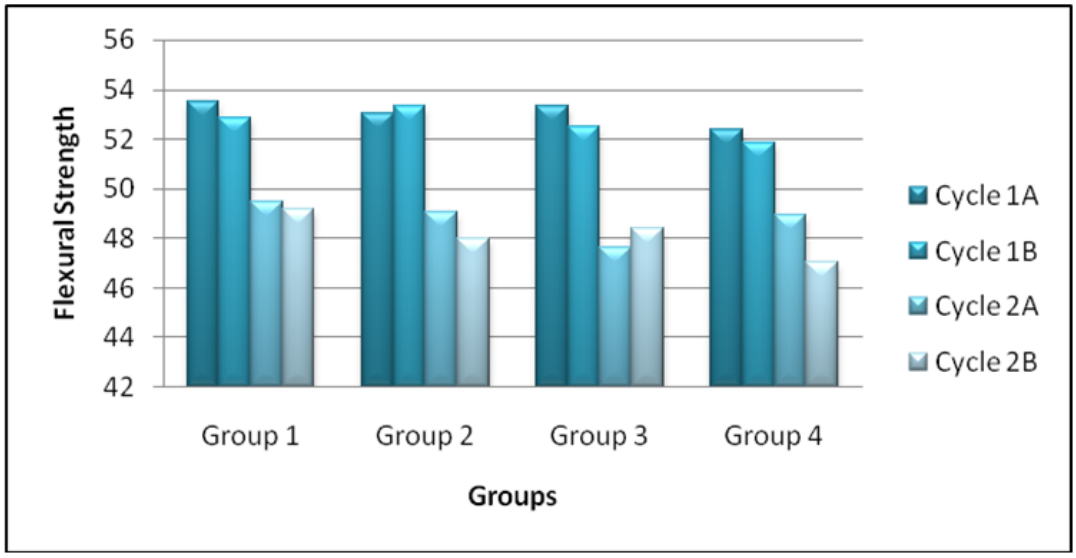

Figure 4. Graph representing flexural strength of the four groups

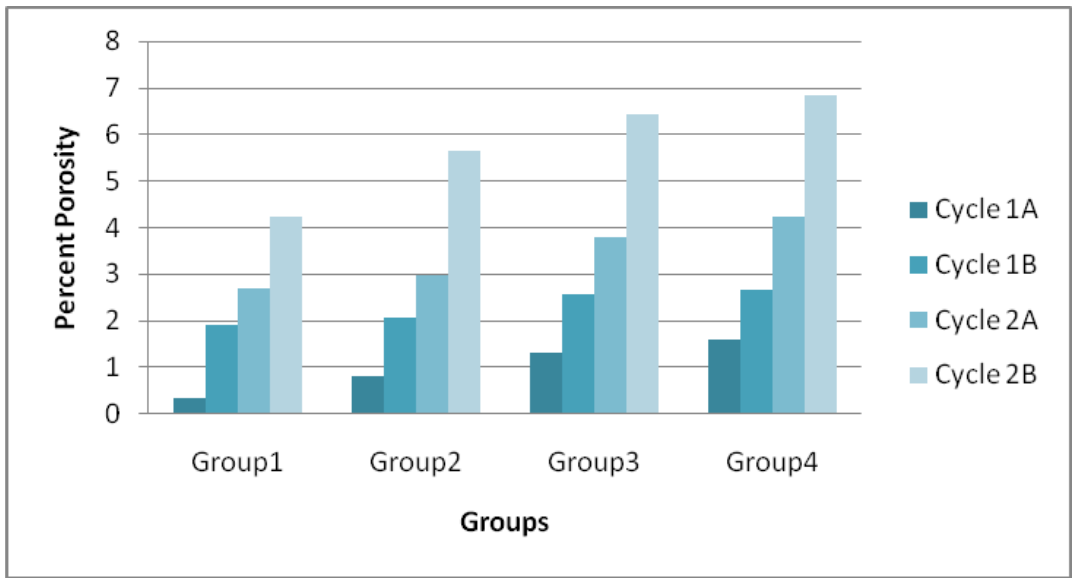

Figure 5. Graph representing the mean percent porosity of the four groups

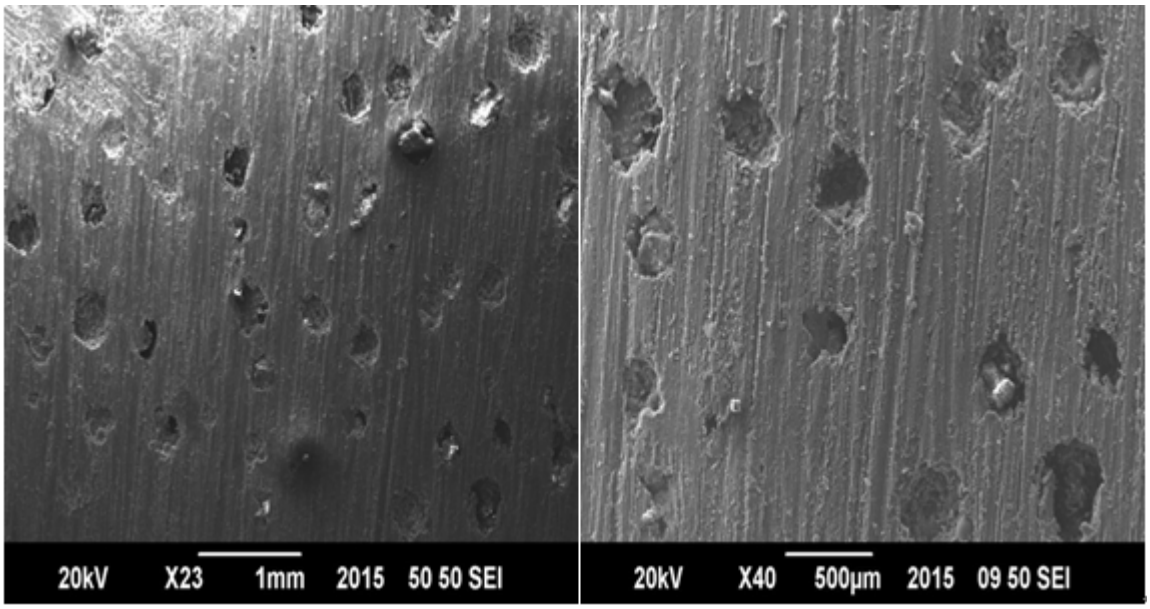

Figure 6. SEM images of specimen cured according to Cycle 1A and1B

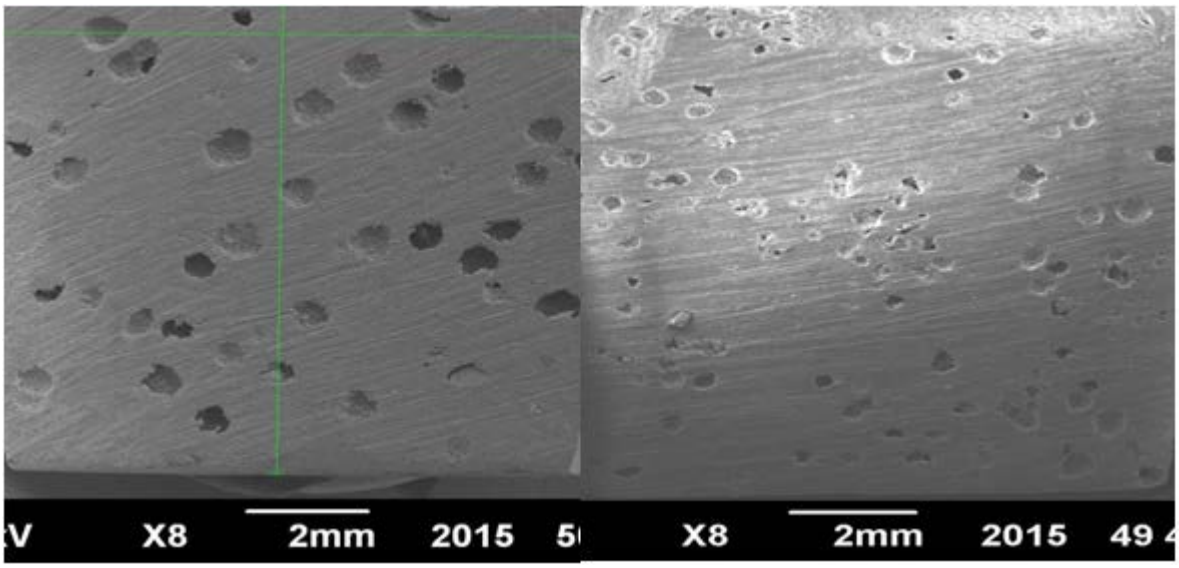

Figure 7. SEM images of specimen cured according to Cycle 2A and 2B

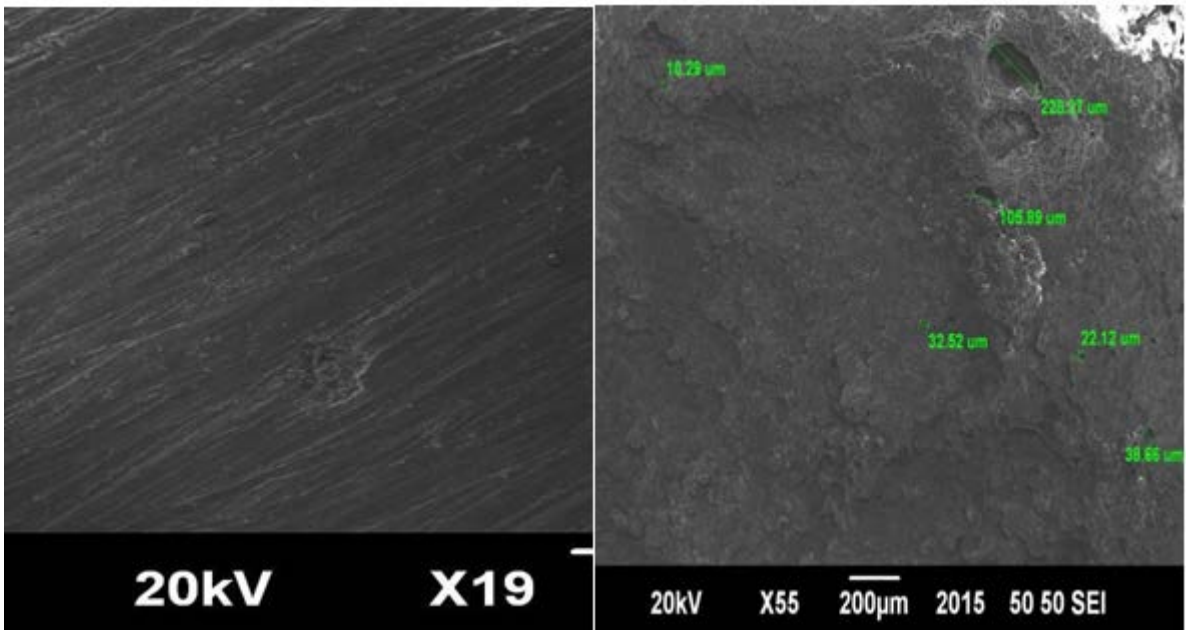

Figure 8. Comparison of surfaces of samples cured by Cycle 1A and 2B

Two sample t test was used to compare the flexural strength and porosity percent values of samples cured using Cycle 1A to those cured with Cycle 2B. The mean flexural strength of samples cured by Cycle 2B (48.16±5.97) was found to be lower than the mean flexural strength (53.08±2.87) of samples cured by Cycle 1A (p value= 0.006), while the mean value of percent porosity of samples cured with Cycle 1A (1.026±0.55) was lower (5.78±1.59) than those cured by Cycle 2B (p=0.00).

The mean flexural strength of samples cured by Cycle 1A (53.08±2.868) was found to be slightly greater than the mean flexural strength (52.64±5.70) of samples cured by Cycle 1B (p value 0.784), while the mean value of percent porosity of samples cured using cycle 1A (1.026±0.55) is less (2.31±0.53) than those cured with cycle 2B (p=0.00). These flexural strength results are not statistically significant and suggest that decreasing the curing time and the terminal boil period by 30minutes in cycles which were started at room temperature, do not affect the flexural strength of the cured acrylic resin specimens. On the other hand the results of percent porosity values are statistically significant indicating that altering terminal boil period by 30 minutes effects the mean percent porosity in a cured sample.

4. Discussion

Fracture of denture base resins due to fatigue as a consequence of intermittent yet prolonged flexural stresses is a commonly seen mode of clinical failure [15,16]. The three-point bend has been considered useful in comparing flexural strengths of denture base materials [17,18]. The flexural strength of the cured specimens in this study were measured in a universal testing machine by the short beam three-point bend test [14]. Various researchers have used different time periods of underwater storage, however, in this study the specimens were stored at 37°C for 28 days, as studies have shown that the influence of water on the flexural strength occurs during the first four weeks of water immersion and longer periods of immersion do not

show a statistically significant decrease in flexural strength [18,19].

Invasive as well as non-invasive techniques have been mentioned in literature for porosity measurements. The Archimedes method of measuring porosity [7,20,21] is considered an easy noninvasive technique, however the use of SEM micrographs allows a higher level of precision evaluation of the specimen pores hence appears to be a more reliable method [22,23].

A moderately negative correlation (-0.394) exists between flexural strength values and the curing cycles, indicating that the flexural strength decreases from cycle 1A to cycle 2B. These results suggest that long curing cycles along with a terminal boil would result in enhancing flexural strength values, hence improving the mechanical properties of the polymer [18]. This is also supported by a strong positive correlation of percent porosity (0.873) with the curing cycles suggesting that porosities increase from cycle 1A to cycle 2B. Presence of increased amount of porosities effects the strength of the acrylic resin specimen as it concentrates stresses in the matrix and contributes to formation of micro-cracks under loading [24]. Porosities also present an air- inhibited layer of incompletely polymerized matrix at the void surface [3,4]. The mechanical properties of heat cured acrylic resins are closely related to curing conditions and are affected by the level of residual monomers [9]. Studies have demonstrated that including a terminal boiling period to the curing cycle produced specimen with low levels of residual monomers and greater flexural strength as compared to specimens cured without a terminal boil [3,25,26]. SP Kasina et al compared the porosities of acrylic denture bases processed by conventional heat polymerization and microwave technique and concluded that a conventional heat polymerization with a terminal boil produces less porosity which depends not only on the polymerization method but also on the type of acrylic resin used [21]. A terminal boil ensures a thorough polymerization and starting the curing cycles from room temperature allows more time for monomer- polymer interaction as well as prevents premature vaporization of monomer.

The decomposition of benzoyl peroxide is temperature dependent, hence in long curing cycles greater decomposition of benzoyl peroxide would result in greater number of polymer chains. Conversion of monomer to polymer is also time dependent and the rate of conversion is greatly increased by increasing the temperature from 70°C to 100°C. This would explain higher flexural strength and lower percent porosity values of samples cured according to cycle 1A and 1B in which flasks were kept in a water bath at room temperature and the temperature was gradually increased, followed by a terminal boil of 60 and 30 minutes respectively.

A rapid polymerization cycle (Cycle 2B) is quite often used to save time, however, it has been demonstrated that such cycles are inefficient in producing complete polymerization and reducing residual monomer levels which is an important parameter in determining mechanical properties [13,27].The un-reacted monomers serve as plasticizers, which reduce strength by a decrease in interchange forces, making deformation to occur more easily under load [28,29]. Cycle 2B in which the curing assembly was placed in the water bath at 100°C showed the least meanflexural strength and highest percent porosity levels, due to a total heat being generated higher than the boiling point of the monomer causing development of internal porosity which in-turn affects mechanical properties [30,31].

K.Z.Islam et al cured acrylic resin specimens at 100°C for 20, 40 and 60 minutes and found no statistical difference among the flexural strength of the cured specimens [32].This is in agreement with our study in which the samples cured according to cycle 2A which involves immersing the flasks in a water bath at a temperature of 70°C followed by gradually increasing the temperature to 100°C and finally maintaining it for 30 minutes and cycle 2B which involves placing the flask directly in a water bath at a temperature of 100°C and for 30mins, had no statistically significant difference ($p=0.740$) in the flexural strength values.

Powder-liquid ratio of PMMA resins is often changed to modify handling properties, however, it was noted that this may have deleterious effects on the properties of the cured material [33]. In this study the acrylic specimens were made according to three different powder-liquid ratios while specimens in group 4 were prepared by final year undergraduate students who dispensed and mixed the powder and the liquid under supervision. The students mixed the acrylic resin without following any ratio or measuring system, hence it was expected that due to a possibly elevated residual monomer concentration in this group, the flexural strength and the porosity levels of the cured specimen would be reduced. However, the results showed a weak negative correlation (-0.085) between the groups and the flexural strength and a weak positive correlation (0.286) with mean percent porosity of the cured samples. These results indicate that the variations of our powder-liquid ratios have a week effect on the flexural strength as well as the percent porosity values of the acrylic resin specimens as compared to the effect of curing regimes. Although this is in agreement with a study carried out by G Greets et al [34], who concluded that powder-liquid ratio does not have a significant effect on the flexural strength of unreinforced PMMA resin, the powder-liquid variations chosen in our study are only slightly different than the manufacturer recommended powder-liquid ratios. A gross change in powder-liquid ratio would undoubtedly result either the mix being too dry and consequently un-manageable or too weak due to excess liquid monomer.

5. Conclusion

Within the limitations of this study, following conclusions can be made:

1. Variations in polymerization cycles have a higher influence on flexural strength and percent porosity of acrylic resins rather than powder-liquid ratios.
2. Starting the curing cycle at room temperature and gradually increasing the temperature seems to produce acrylic resin specimen with greater flexural strength and low percent porosity values as compared to curing cycles in which the flasks are placed directly at boiling water.
3. Increasing the overall curing time and terminal boil period from 30 to 60 minutes in cycles which started

at room temperature, does not have a significant effect on the flexural strength values; however the percent porosity levels are significantly affected.

4. Large porosities are produced in specimens cured by placing the flasks directly in boiling water, hence lowering the observed flexural strength values.

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