



## **Evaluation of The Effect of Temperature and Time on The Conversion Factor of Acrylic Resin Denture Base Material.**

### **ABSTRACT**

**The aims of this study** were to evaluate the effect of change in time ,temperature (increase or decrease) on the degree of conversion and residual monomer release of the vertex heat cured acrylic resin. **Materials and methods:** The total number of samples (176) were divided into three main groups cured according to ADA and three curing cycles with modifications in time and temperature, degree of conversion and residual monomer properties were measured for all the samples. **Results** were analyzed statistically by (Descriptive statistics, ANOVA and Duncan's multiple range test) and showed that both changes in time and temperature showed significant changes in the degree of conversion, residual monomer. **Conclusion** is that the rapid simplified and regular manufacturer's instruction curing cycles showed the best results regarding the degree of conversion, residual monomer.

### **الخلاصة:**

**الأهداف:** إن الغاية من هذه الدراسة هو تقييم تأثير التغيير في درجة الحرارة ووقت البلمرة (زيادة أو نقصان) على درجة التحول و كمية المونيمر المتبقي لمادة الراتنج الاكريلي المتصلب بالحرارة vertex لقاعدة الطقم . **المواد وطرق العمل:** العدد الكلي لجميع العينات (١٧٦) تم تحضيرها ، هذا العدد قسم الى ثلاثة مجموعات رئيسية صليت حسب دورة التصلب الحرارية القياسية (ADA) مع تحويلات في الوقت و درجة الحرارة لقياس درجة التحول و كمية المونيمر المتبقي لمادة قاعدة الطقم. **النتائج:** وقد تم استخدام التحليلات الإحصائية الأتية: التحليل الوصفي و تحليل التباين (ANOVA) و (Duncan Multiple Range Test). وقد أشارت نتائج الدراسة إلى أن التغييرات في درجات الحرارة والوقت تؤدي الى تغييرات معنوية في درجة التحول, كمية المونيمر المتبقية . **الاستنتاجات:** نستنتج من هذه الدراسة إن أفضل النتائج بالنسبة إلى درجة التحول وكمية المونيمر المتبقية قد أمكن الحصول عليها عند بلمرة مادة قاعدة الطقم حسب طريقة التصلب المفضلة من قبل المصنع.

**Nadira A.Hatim** (Prof.);  
**Dr.Amer A.Taqa** (Assist.  
Prof.)  
**Saja Amjad Abdul Razzak**  
(Assist. Lect. )

*Department of Prosthetic Dentistry  
College of Dentistry, Mosul University*

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The number of denture wearers is increasing as the number of elderly people continually growing, and polymethylmethacrylate (PMMA) is still the most frequently used material in denture base fabrication <sup>(1)</sup>.

The biological characteristics of denture – base resins are influenced by the monomer to polymer conversion. Despite the various methods used to initiate the polymerization of acrylic resins, the conversion of monomers to polymers is incomplete leaving residual monomer in the denture base that can leach into the saliva <sup>(2,3)</sup>.

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Barron *et al.* <sup>(4)</sup> stated that the degree of conversion of resin materials is a measure of the carbon double bonds (C=C) converted into carbon single bond (C-C).

There is an inverse relationship between the degree of conversion and the residual monomer content, thus the higher the former, the lower the later <sup>(5,6)</sup>.

The amount of residual monomer is one of the principal factors affecting the properties of acrylic resin denture bases <sup>(4,7-10)</sup>.

The purposes of this study were to evaluate the effect of change in time, temperature (increase or decrease) on the degree of conversion and residual monomer release of the vertex heat cured acrylic resin.

### MATERIALS AND METHODS

The total number of samples (176) were prepared from vertex heat curing denture base resin material according to manufacturer's instructions powder/liquid mixing ratio (2/1). Samples were divided into three main groups as shown in figures (1,2,and 3).

The Changes or modifications in temperature were controlled by the thermo state of the water bath, while changes in times were monitored by the clock timer, curing at 105°C was obtained by addition of salt to the water bath at a ratio of 20 g salt: 100 ml water <sup>(11)</sup>.

**Degree of Conversion Measurement Test:** Three samples of vertex heat – polymerizing resin with dimensions of 10x4x4 (±0.03) mm were prepared and stored in distilled water at 37±1 °C for 48 hour <sup>(12)</sup>. After 48 hour, the samples were removed from water and dried in air and then scraped using a sharp, clean and sterile wax knife, then 1 – 2 mg of the sample powder was grinded finely, under anhydrous conditions, in an agate mortar. This powder is then thoroughly mixed with 100 – 200 mg of oven – dried, spectral – grade, 100 – 200 mesh potassium bromide powder. The mixture is then pressed into a transparent pellet by using a mini – press. The KBr pellet is then mounted on a holder and placed in the sample beam of the infrared spectrophotometer <sup>(13)</sup>.

In the IR charts, two absorbance peaks were determined (the absorbance peak of the C=C from the methacrylate group, and the absorbance peak of the C=O from the ester group). An appropriate base line was drawn, the intensity of absorption was determined by calculating the areas of the peaks by the Auto CAD program <sup>(14)</sup>. The degree of conversion was calculated from the following formula <sup>(12)</sup>:

$$DC\% = \left[ 1 - \frac{[\text{Abs}(C=C) / \text{Abs}(C=O)] \text{ polymerized specimen}}{[\text{Abs}(C=C) / \text{Abs}(C=O)] \text{ monomer}} \right] \times 100\%$$

**Residual Monomer Measurement Test:** Five Samples with linear dimensions 20x20x3(±0.03) mm <sup>(15)</sup> were prepared for each cycle. After curing, each sample was introduced in sealed glass flask containing 10 ml of distilled water at 37°C. At appropriate times (1<sup>st</sup>, 2<sup>nd</sup>, 3<sup>rd</sup>, 4<sup>th</sup>, 5<sup>th</sup>, 6<sup>th</sup> and 7<sup>th</sup> days), the supernatants were removed and replaced by 10 ml of fresh distilled water. The time – dependence of the monomer concentration was followed by monitoring the amount of monomer present in the supernatant medium using a CECIL 2000 UV – Visible Spectrophotometer (λ= 254 nm). A linear calibration curve of methylmethacrylate (MMA) concentration as a function of the absorbance at 254 nm was obtained using MMA standard aqueous solutions in the range 0.025 – 0.5 mg/ml (Figure 4). The results were expressed as a percent of released residual monomer mass with respect to the

weight of the specimen<sup>(15)</sup>. Statistical analysis used in this study were Descriptive statistics, ANOVA and Duncan's multiple range test.

## RESULTS

**1.The Degree of Conversion Test:** IR charts (Figures 5,6), two absorbance peaks appeared (the absorbance peak of the C=C from the methacrylate group at  $1640\text{ cm}^{-1}$  and the absorbance peak of the C=O from the ester group at  $1720\text{ cm}^{-1}$ ). By using Auto CAD program, the results were calculated by one way analysis of variance (ANOVA) for the degree of conversion test (Table 1) showed that there were significant differences ( $p<0.05$ ) between the three standard curing cycles and their modification groups in comparison with the standard short cycle .

Mean, standard deviation and Duncan's multiple range test for the standard short cycle S1 (ADA) and its modification groups (Figure 7) indicated that all the modification groups showed significant increase in the degree of conversion from the standard short cycle S1 (ADA), except groups S2 and S4 which showed significant decrease in the conversion from the standard cycle (ADA). Both of these groups represent decrease in the curing temperature. While groups S6 and S8 which represented decrease in the curing time showed no significant difference from the standard cycle.

Duncan's multiple range test of the degree of conversion test of the standard short curing cycle (S1) with the rapid simplified and regular manufacturer's instructions curing cycle and their modifications (MRa) and (MRe) groups indicated that the standard short curing cycle (ADA) showed significant decrease in degree of conversion from the standard rapid simplified and regular heat curing cycles and all their modifications ,as shown in Figures (8 and 9).In comparison of the three standard curing cycles, it is shown that the rapid simplified had the highest conversion followed by the regular, then the standard short cycle(Figure 10).

**2. Residual Monomer Release Test:** The amount of released monomer versus release period of time (in days) for the short cycle, rapid simplified and regular heat curing cycles and their modifications was illustrated in Figures (11-13).

## DISCUSSION:

**1.The Degree of Conversion Test:** It is shown from the results (Figure 7) that group (S3) showed significant increase in the degree of conversion from the control S1, groups MRa3,MRe3 showed not significant decrease in degree of conversion from their standard MRa1, MRe1 cycles (Figure 8-10) and significant increase in the degree of conversion in comparison with the standard ADA These cycles represented curing with increase of the first temperature of the curing cycle .This increase in conversion with temperature increase was stated by Beech, Bartoloni *et al.*, and Lovell *et al*<sup>(2,16,17)</sup>. The reasons for increased conversion are based on many factors, increased temperature decreases system viscosity, the collision frequency of un-reacted active groups and radicals increases with elevated curing temperature when below the glass transition temperature . Furthermore, as temperature is raised, additional free volume increases, giving trapped radicals increased mobility, resulting in further conversion<sup>(18)</sup>.

Groups (S4, MRa4 and MRe2) showed significant decrease in the degree of conversion in comparison with their control groups S1, MRa1, MRe1. These groups were cured by lowering the boiling point, this indicates that reaching boiling point is necessary for increased conversion and this agrees with Harrison and Hugget,<sup>(19)</sup> as they stated that it has been recommended that the polymerization cycle should include a terminal boiling treatment for at least one hour to achieve maximum monomer conversion. This finding also agrees with many

authors<sup>(12,20,21)</sup>. Groups (S5&MRe3) showed increase in the degree of conversion with increasing the boiling point to (105°C). This may be explained that, as the amount of monomer is reduced as a result of the polymerization, it becomes more difficult to bring the monomer and the free radical together, because the available heat stabilizes. The more the temperature rises, the faster the molecules move and the more complete is the polymerization reaction<sup>(7)</sup>. Groups (S7 and MRe5) showed increase in conversion with increasing the curing time from their control groups. Authors<sup>(15)</sup> showed that it is possible to optimize the conversion degree by varying the time of curing at constant temperature, while Beech<sup>(16)</sup> stated that long reaction times increase the polymer yield.

Groups S9 and MRe5 showed increase in conversion respectively in comparison with the control. These groups cured at prolonged boiling cycle<sup>(6,19)</sup>.

**2. Residual Monomer Release Test :** The highest amount of residual monomer released was observed in group S4 which is cured by modification of the curing temperature by lowering the boiling point, while group S5 showed lowered residual monomer than S1 (cured by increasing the boiling temperature). Group S7 also showed low residual monomer release (Figure 11-13). This group cured by modification in the curing time (increase), all these results come in agreement with Mohamed *et al.*<sup>(22)</sup>. As they showed that increased temperature and extended polymerization time were accompanied by a decrease in the residual monomer content. Groups S9 and MRa8 showed low residual monomer release, these groups cured by increasing the boiling time, this comes in agreement with Dogan *et al.*<sup>(23)</sup> they showed that longer curing times at 100°C decreased the level of residual monomer.

All groups showed greatest loss in the first 24 hours, this agree with many authors<sup>(24,25)</sup> After 48 hour, the content of monomer release were very low, then gradual decrease in the remaining monomer release until reach zero. Different mechanisms might help explain this reduction; it has been observed that the concentration of the residual monomer in the polymerized resin can be diminished by diffusion into water and by continuous polymerization promoted by the active radicals found in the polymer chains<sup>(5,2)</sup>

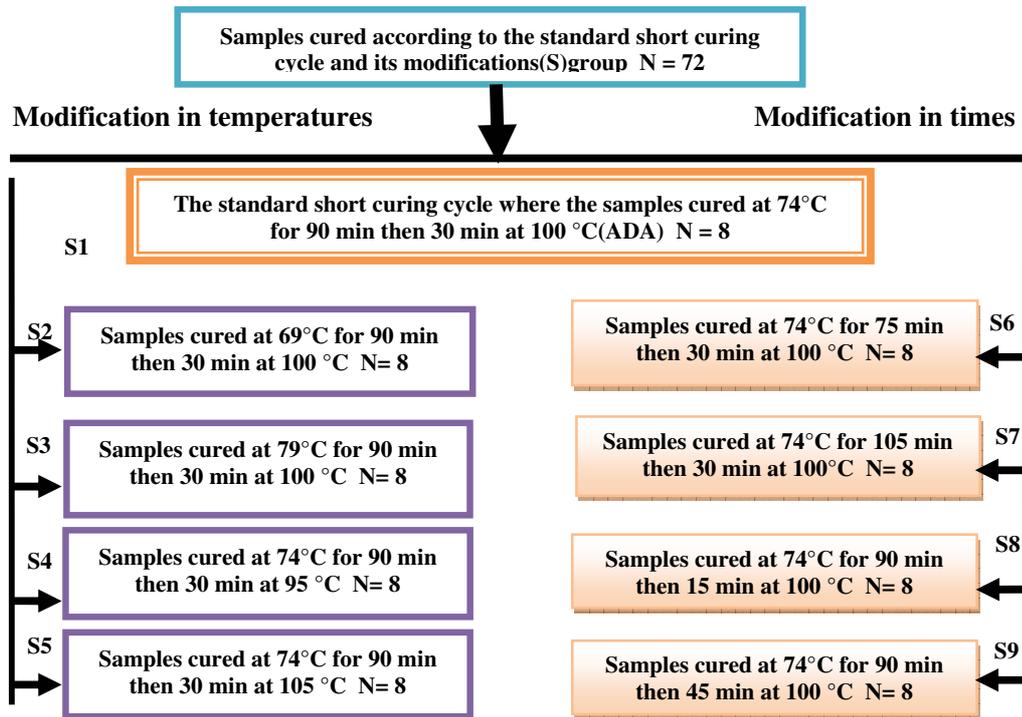


Figure (1) The distribution of the samples of the short cycle (ADA) and its modifications (S)group.

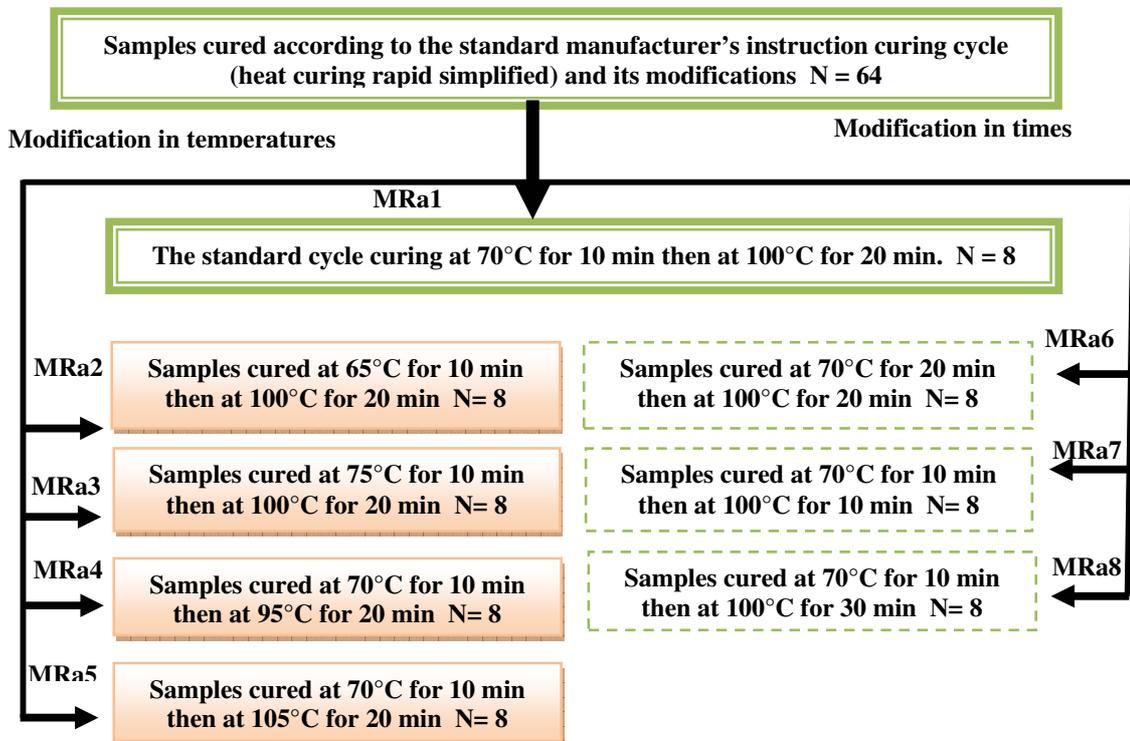


Figure (2): The distribution of the samples of the rapid simplified manufacturer's instructions curing cycle and its modifications, (MRa) group

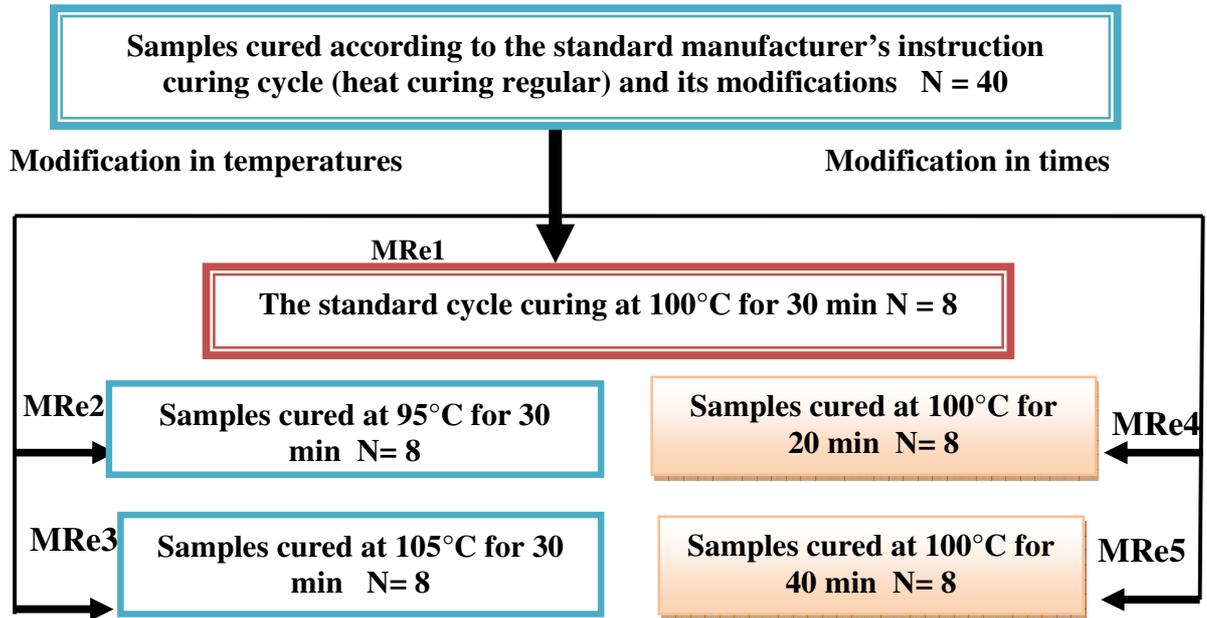


Figure (3): The distribution of the samples of the regular manufacturer's instructions curing cycle and its modifications, (MRe) group

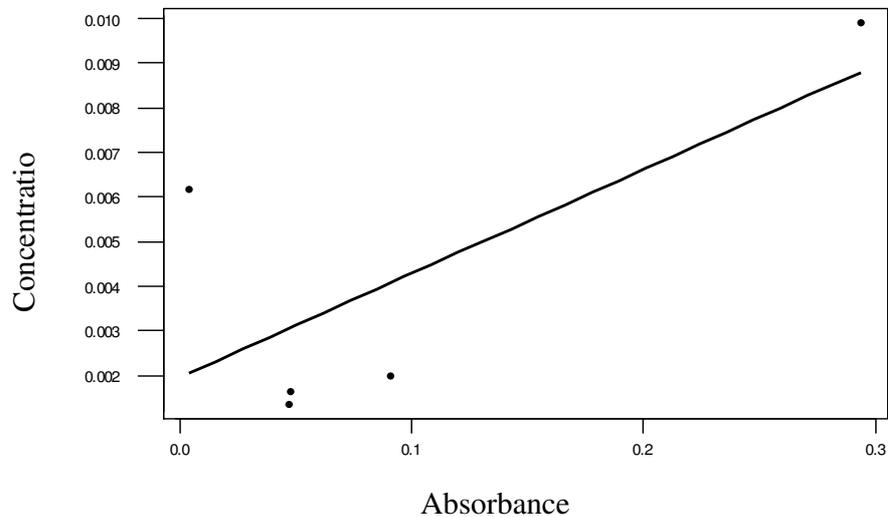


Figure (4) Calibration curve of monomer.

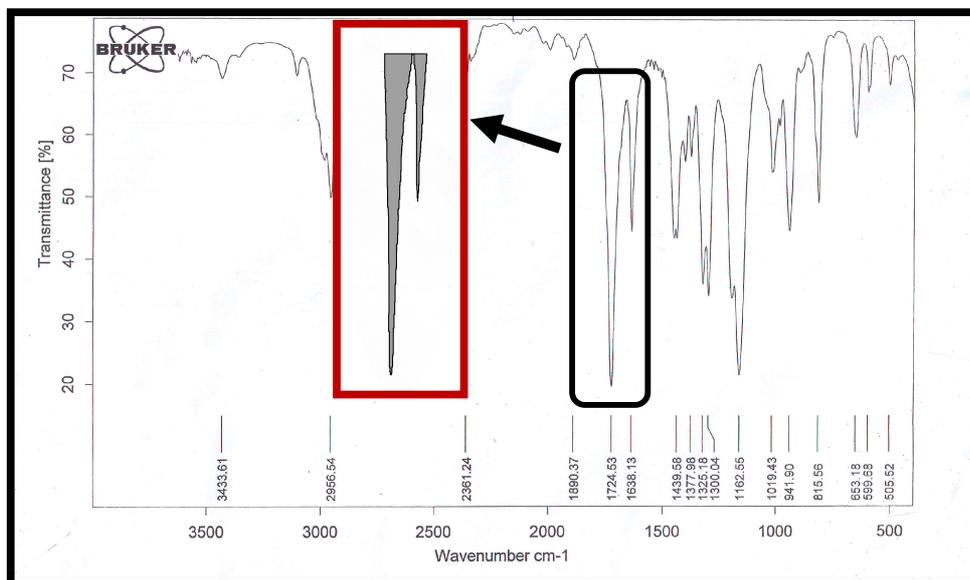


Figure (5): Infrared spectrometer chart of the monomer (methylmethacrylate)

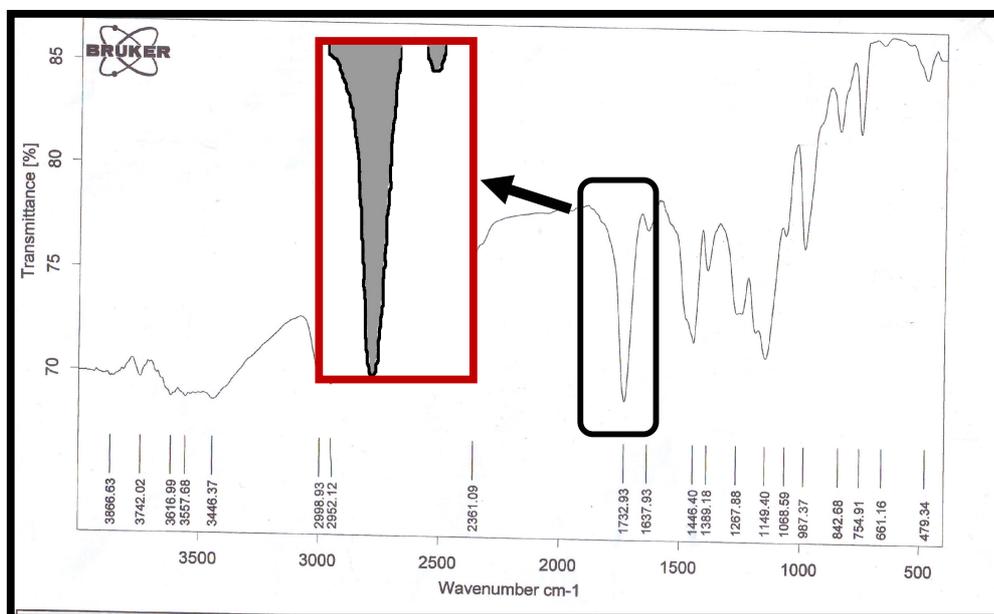


Figure (6): Infrared spectrometer chart of the sample prepared according to the standard short cycle (ADA)

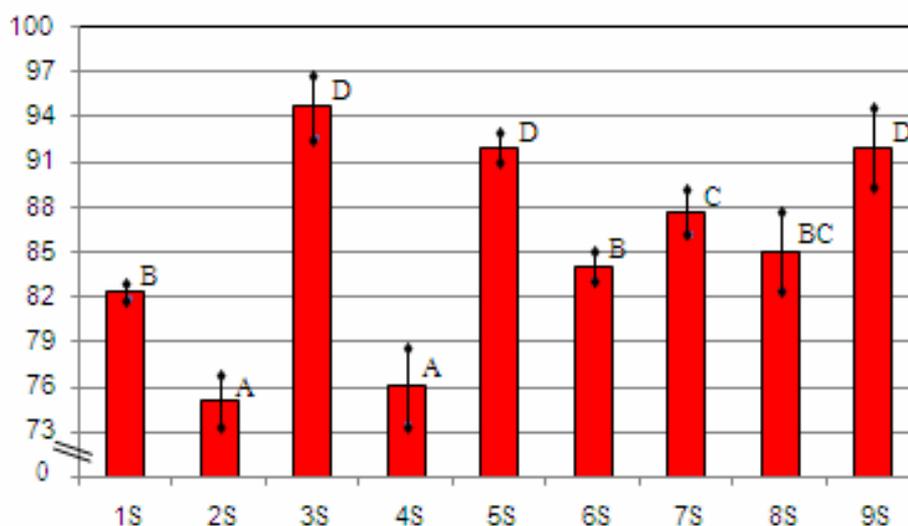


Figure (7): Mean  $\pm$  SD and Duncan's Multiple Range Test of the degree of conversion test for the short cycle and its modification groups (S) group

\* Different letters mean significant difference at  $p \leq 0.05$ .

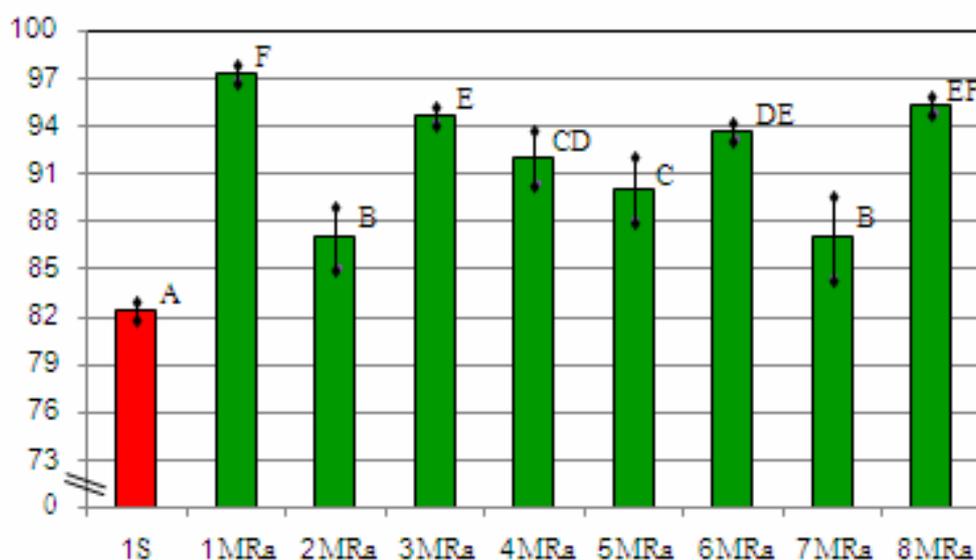


Figure (8): Mean  $\pm$  SD and Duncan's Multiple Range Test of the degree of conversion test for the rapid simplified heat curing cycle and its modifications in comparison with the standard short cycle

\* Different letters mean significant difference at  $p \leq 0.05$ .

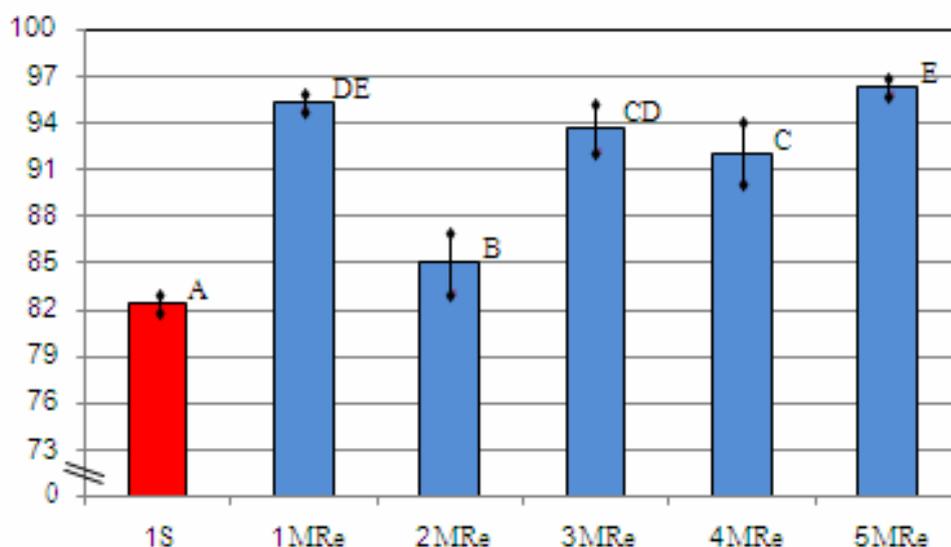


Figure (9): Mean  $\pm$  SD and Duncan's Multiple Range Test of the degree of conversion test for the regular heat curing cycle and its modifications (MRa) group and the standard short cycle  
 \* Different letters mean significant difference at  $p \leq 0.05$ .

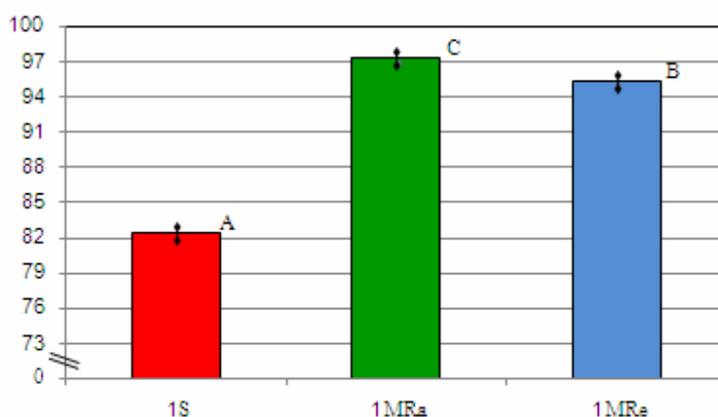


Figure (10): Mean  $\pm$  SD and Duncan's Multiple Range Test of the degree of conversion test for the three standard curing cycles (short curing cycle, rapid simplified and regular heat curing cycles)  
 \* Different letters mean significant difference at  $p \leq 0.05$ .

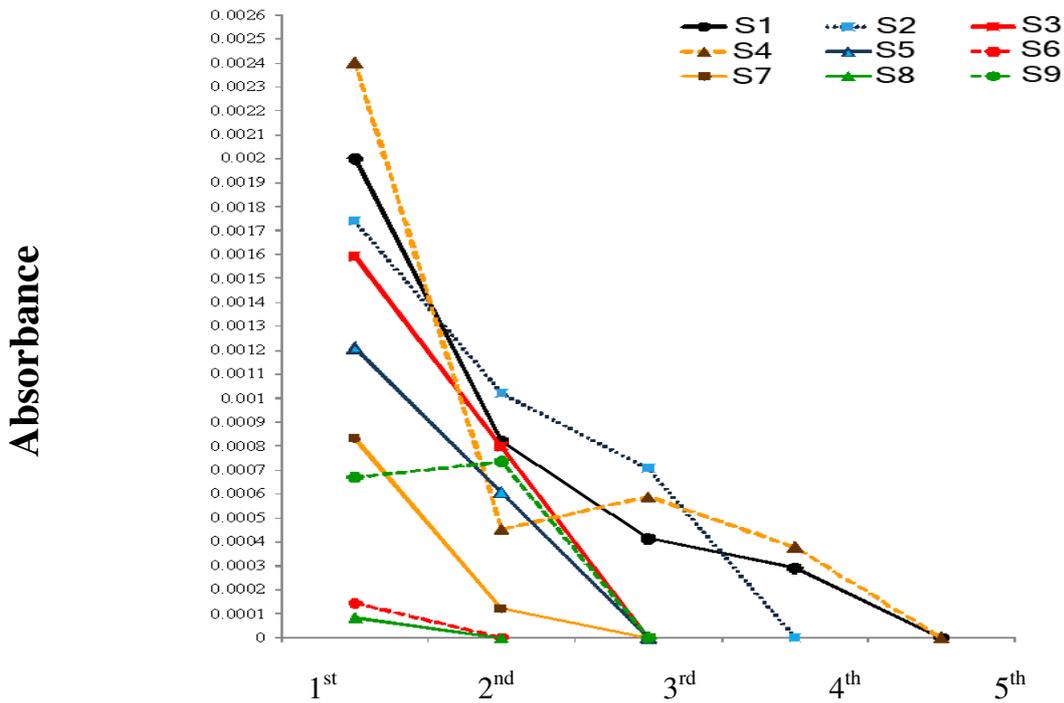


Figure (11) The residual monomer release of the short cycle and its modification groups .

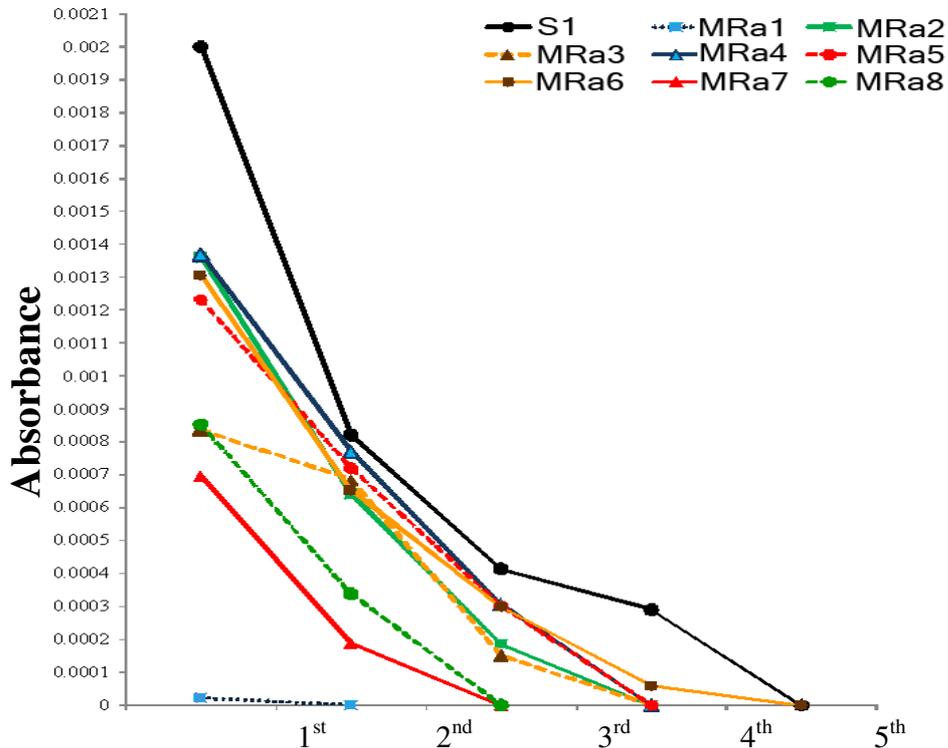


Figure (12) The residual monomer release of the rapid simplified curing cycle and its modification groups in comparison with the standard short cycle (ADA).

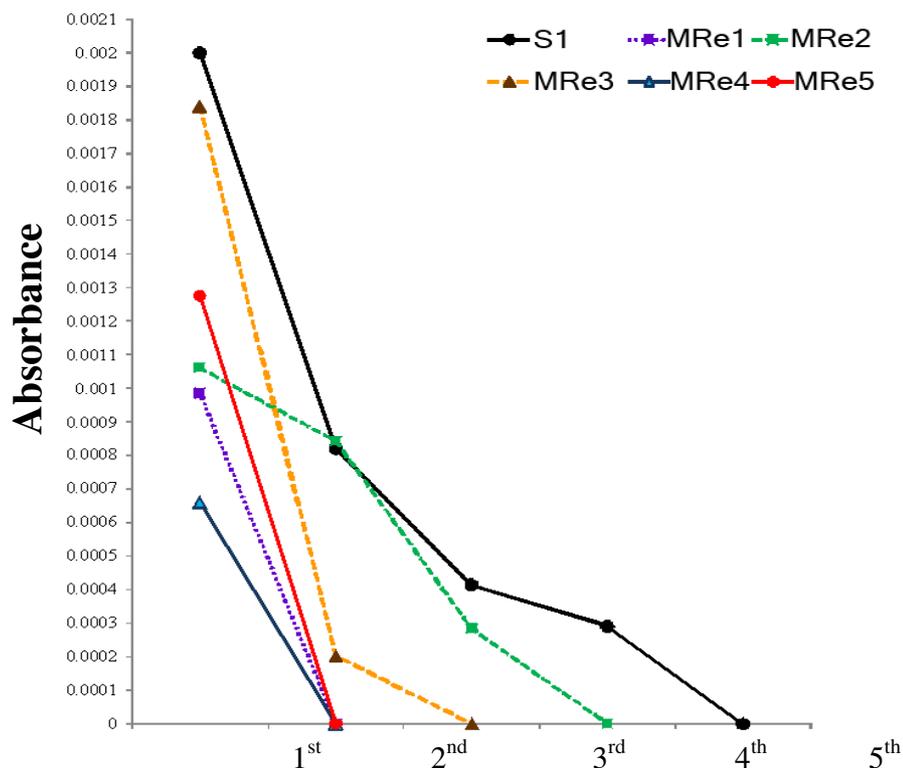


Figure (13) The residual monomer release of the regular heat curing cycle and its modification groups in comparison with the standard short cycle (ADA).

Table (1) The one way analysis of variance (ANOVA) of the degree of conversion test For the short curing cycle, rapid simplified and regular heat curing cycles and their modification groups(without additives).

Groups		Sum of squares	df	Mean square	F	Sig.
S1→S9	Between groups	1158.519	8	144.815	39.495	0.000*
	Within groups	66.00	18	3.667		
	Total	1224.519	26			
S1 with MRe1→MRe8	Between groups	565.630	8	70.704	32.356	0.000*
	Within groups	39.333	18	2.185		
	Total	604.963	26			
S1 with MRe1→MRe5	Between groups	498.444	5	99.689	52.776	0.000*
	Within groups	22.667	12	1.889		
	Total	521.111	17			

\* Significant difference at p<0.05. df:Degree of freedom.

## CONCLUSIONS

1. Curing the resin by following the manufacturer's instructions curing cycle of the supplied resin (At 70 °C for 10 minutes then at 100°C for 20 minutes or at 100°C for 30 minutes) provided the optimal properties regarding the highest conversion and lowest amount of released residual monomer.
2. Modifications in the curing temperature showed more significant effect on the studied properties than modifications in time.
3. In general, raised temperature and extended polymerization time showed improved conversion ,and lowering the monomer release
4. Increasing the time at boiling during curing for the three studied curing cycles resulted in improvement in the degree of conversion

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