Strength and Surface Roughness of Cross Linking Acrylic Resin Processed by Different Heat Curing Methods

*Ali Ehsan Kareem**

B.D.S., M.Sc.(prosthodontics), Assist. Lec. College of Dentistry - Al-Mustansirya University

ABSTRACT

Background: Heat-cured acrylic resin is considered one of the most commonly used denture base material in denture construction, but this material is not ideal in every respect. So many researches have been carried out to find an alternative material like cross linking acrylic resin. Meanwhile, the curing procedures have been modified to improve the physical and mechanical properties of resin materials .The aim of this study was to evaluate the effect of three curing methods:(water bath, autoclave short cycle and autoclave long cycle) on transverse strength, tensile strength and surface roughness of cross linking acrylic denture base materials.

Material and method: Cross linking acrylic resin was used in this study and processed in three curing methods (water bath, autoclave short cycle and autoclave long cycle). Ninety specimens were prepared. Each main group was subdivided into three subgroups according to the type of test used (transvers strength test, tensile strength test and surface roughness test). Each group contained 10 specimens for each test.

Result: Three tests were used to analyze the results: analysis of variance (ANOVA), the least significant difference (LSD) and an independent T-test. There were no significant differences between the results of the processing techniques regarding transverse and tensile strength but there was a significant difference between the results of surface roughness tests.

Conclusion: It was concluded that autoclave curing method can be used with different duration whether it is short or long instead of water bath curing method.

KEY WORDS

Cross linking acrylic resin, autoclave, water bath, strength, roughness.

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القوة وخشونة السطح لراتنج الاكريلك المتشابك والمتبلمر حراريا بطرق مختلفة

علي احسان كريم

مدرس مساعد/ كلية طب الاسنان- الجامعة المستنصرية ماجستير صناعة الاسنان

المستخلص

يعتبر راتنج الاكريلك المتبلمر بطريقة البلمرة الحرارية باستخدام الحمام المائي هو احد المواد الاكثر استخداما كقاعدة طقم الاسنان لكن هذه المادة ليست مثالية في جميع النواحي, وقد اجريت الكثير من الابحاث لايجاد مادة بديلة مثل راتنج الاكريلك المتشابك. وفي الوقت نفسه تم تعديل اجراءات البلمرة الحرارية لتحسين الخواص الفيزيائية والميكانيكية لمواد قاعدة طقم الاسنان. وكان الهدف من هذه الدراسة هو تقييم تاثير ثلاث طرق بلمرة: (بلمرة بواسطة حمام الماء, بلمرة بواسطة جهاز التعقيم البخاري لفترته القصيرة والطويلة) على القوة المستعرضة, وقوة الشد وخشونة السطح لراتنج الاكريلك المتشابك المستخدم كقاعدة لطقم الاسنان.

لقد تم استخدام راتنج الاكريليك المتشابك في هذه الدراسة وتم بلمرته بثلاث طرق بلمرة: بواسطة حمام الماء, بواسطة دورة قصيرة لجهاز التعقيم البخاري وبواسطة دورة طويلة لجهاز التعقيم البخاري. لقد تم اعداد ٩٠ عينة وتم تقسيم كل مجموعة رئيسية الى ثلاث مجموعات فرعية وفقا لنوع الاختبار المستخدم (اختبار القوة المستعرضة, اختبار قوة الشد واختبار خشونة السطح). احتوت كل مجموعة على ١٠ عينات لكل اختبار.

النتائج: تم تحليل النتائج بواسطة ثلاثة اختبارات LSD ، ANOVA، واختبار T المستقل. لا توجد فروق ذات دلالة إحصائية بين نتائج تقنيات البلمرة فيما يتعلق بالقوة المستعرضة وقوة الشد ولكن كان هنالك فرق كبير بين نتائج اختبار خشونة السطح.

الخلاصة: البلمرة بواسطة جهاز التعقيم البخاري سواء كانت قصيرة أو طويلة يمكن استخدامه بدلا من البلمرة بواسطة حمام الماء.

الكلمات الرئيسية: راتنج الأكريليك المتشابك، جهاز التعقيم البخاري، حمام الماء، القوة، خشونة السطح.

INTRODUCTION

Poly(methyl methacrylate) (PMMA) is a derivative of acrylic acid, referred to as acrylic resin and it was introduced for use in dentistry in the early 1930s. Since that time it became the most reliable material for denture construction.(1,2)Today the vast majority of dentures made are fabricated from heat-cured PMMA and rubber-reinforced PMMA.(3)All denture bases constructed from PMMA polymers or copolymers.(4)

This material is not ideal in every respect and it is in combination of various properties rather than one single desirable property that accounts for its popularity and usage. The properties contributed to the success of acrylic resin as a denture base material are excellent appearance, ease in processing, simple processing equipment and ease repair.(5,6,7) In addition, it is characterized by low cost of fabrication, a satisfactory shelf life. (8, 9, 10)But it does not provide sufficient mechanical requirements of the dental prosthesis.(11) Due to its residual monomer evaporation, this results in polymerization shrinkage, dimensional inaccuracy, rough surface, and porosity. This material has limitation, particularly in term of

*: E- mail: ali_ihsan123@yahoo.com

flexural and impact strength.[5,6] Also, Johastone et al. showed that 68% of acrylic resin dentures break within few years after fabrication[12].

Different trials had been undertaken by many researchers to improve the mechanical properties of PMMA,[6]Such as, reinforcement of different types of fibers[11], metal wires were also used as reinforcement for denture base polymers[13] and the incorporation of fillers, search for an alternative material to PMMA, use of chemical modification, presence of surface treatment or not and using alternative curing method or changing the programed cycle. Strong and more fracture-resistant denture base materials can be obtained by the use of impact modifiers; usually rubber like substances which were added to acrylic resin [14]or by the addition of cross linking agents.[15] Glycol dimethacrylate is the type of cross linking agent used in PMMA denture base resin. The maim advantage of using cross linking agent is to increase the resistance of polymer to minute surface cracking or crazing. [16]

The addition of about 2-14% cross linking agent improves the resistance of acrylic resin to surface cracking, while the addition of up to 25% have little effect on tensile strength, transverse strength or hardness of acrylic plastic.[17]

Over the years, curing procedures have been modified with a view to improve the physical and mechanical properties of resin materials. Different polymerization methods have been used: heat, light, chemical and microwave energy.[18]Various methods were presented for curing acrylic resin by heat and the most popular method is water bath.[14] Some of the advantages of this methods are simple and low cost but the major disadvantage is long processing time required.[19]

The use of a pressure cooker for denture polymerization was first reported by Muley in 1976.[20]Indian researchers extensively investigated the pressure cooker polymerization technique. Conventional acrylic resin material was used in this technique and required less than 1h for polymerization and utilized conventional equipment.[21]

PMMA denture base resins and their curing processes have been modified not only to improve the physical and mechanical properties but also to improve the working properties.[22,23]

The autoclave processing technique is represents a better substitute for zwater bath technique. Regarding autoclave processing technique, the slow (long) curing cycle provides better denture bases material including the tested physical and mechanical properties as compared with the fast (short) curing cycle.(24)

This study was intended to investigate the effect of water bath and autoclave curing methods on transverse strength, tensile strength and surface roughness of cross linking acrylic denture base materials and to study the effect of time durations of autoclave (short and long) processing technique on these tests.

MATERIALS AND METHODS

The acrylic material used in this research was cross linking acrylic denture base material (SLEDGEHAMMER, KEYSTONE INDUSTRIES/ USA) and it was processed by two different heat curing methods water bath and autoclave.

Ninety specimens were prepared and grouped into: the control group (W) in which acrylic resin processed by conventional water bath processing technique at 74°C for 1.5 hours then boiled for 30 minutes and the experimental groups (A) in which acrylic resins processed by autoclave at 121°C, 210 KPa (Kilopascal). The experimental groups were divided into autoclave short (AS) for 15 minutes and autoclave long (AL) for 30 minutes to study the effect of the autoclave processing, and three tests were conducted: transverse strength, tensile strength and surface roughness tests. For each test 10 specimens were used.

Metal patterns were constructed by cutting a stainless steel plate of 2.5mm in thickness into the desired dimensions according to the requirements of each test, as follows:

The metal patterns were constructed for transverse strength test and surface roughness test with dimensions of (65mm x 10mm x 2.5mm) length, width and thickness respectively [25], and for tensile strength test with dimensions of (65mm X 12.5mm X 2.5mm) length, width, and thickness respectively and the constricted part with 5mm in width. [26]

The conventional flasking technique for complete dentures was followed in mold preparation for each sample. Each metal pattern was coated with petroleum jelly and immersed in the slurry stone (TYPE III HARD STONE, THIXOTROPIC, Zhermach/ Italy), which was prepared according to the manufacturer's instructions and poured into the lower half of the dental flask as in (Figure 1,2). The set lower half was coated with a separating medium(DIVOSEP, VERTEX-DENTAL/Netherlands) and allowed to dry and another layer of stone was poured into the second half of the flask and allowed to stand for one hour then the flask was opened and the metal block was removed.

The metal patterns were then removed from the flask being packed and the two halves of the mold were coated with a separating medium before packing with cross linking acrylic (SLEDGEHAMMER, KEYSTONE INDUSTRIES/USA). The material was mixed and manipulated according to the manufacturer's instructions (3:1) by volume and left under pressure 20 bar (100 Kilopascal (KPa) for 5 minutes before clamping was done. Curing for the control group (W) was carried out by placing the clamped flask in a water bath and processed by short curing cycle 90 minutes at 74C° then temperature was increased to the boiling point 100°C for 30 minutes.[25]

The curing for the experimental group (A) was carried out by placing the clamped flask in a fully automatic autoclave (SW 22 PLUS, STERN WEBER/ Italy) as shown in Figure (3) and processed by the preprogrammed cycles of two durations, as follow:(AS):Short 121ºC/210KPa, 15 min. and (AL):Long 121ºC/210KPa, 30 min. In this research, only two standard programs (short 121ºC & long 121ºC) were used for autoclave curing cycles.

The stages of operation of autoclaves included air removal, steam admission and sterilization cycle including heating up, holding/exposure, and cooling stages.[27,28]The autoclave was operated to start heating the water**,** then the temperature and pressure were raised till they reached (121ºC & 210 Kilopascal) respectively**.** When the temperature reached (121 ºC), the temperature and pressure were held automatically at (121 ºC and 210 KPa respectively for15 minute for short curing cycle and for 30 minutes for long curing cycle ,then automatically exhausted the steam and the programmed cycle was finished. Finally, the metal flask was allowed to cool at room temperature for 30min., followed by complete cooling of the metal flask with tap water for15 min. before deflasking. The acrylic patterns were then removed from stone mold.[4]

Figure (1) Metal pattern for transverse strength and surface roughness test in dental flask

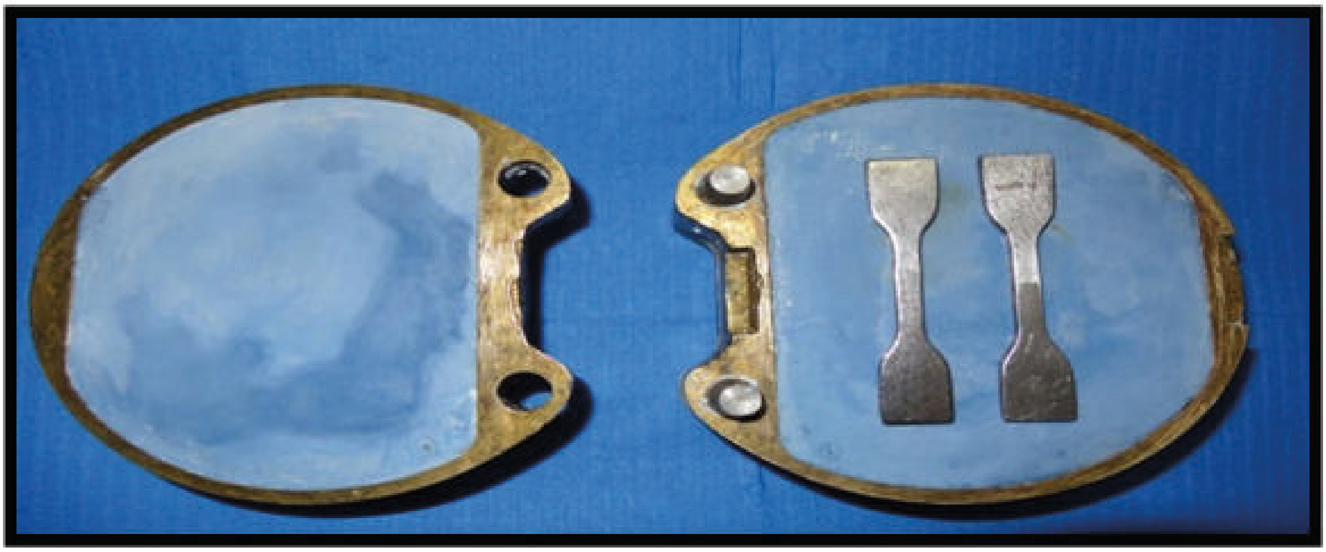

Figure (2) Metal pattern for tensile strength test in dental flask

All the specimens were carefully de-flasked and finished except the specimens used for surface roughness test were not polished. All samples were placed in distilled water in an incubator for 48 hours at a temperature of 37 ºC before they were tested.[25]

Mechanical and physical tests:

1. Transverse strength test:

The test was carried out by using the instron testing machine (Figure 4), each specimen was positioned on bending fixture, consisting of 2 parallel supports of 3.2mm diameter placed (50) mm apart. The full scale load was 50kg and the load was applied with cross head speed of 1mm/min by rod placed centrally between the supports making deflection until fracture occurred.

The transverse bend strength was calculated in N/mm² using the following formula:

$S=3PL/2bd^2$ [29]

Where

S: Transverse strength N/mm² (Newton per square millimeter) or MPa (Megapascal)

P: is the peak load in Newton, L: is the span length in millimeter, b: is the sample width in millimeter, d: is the sample thickness in millimeter.

2. Tensile strength test:

The test was carried out by using the instron testing machine with grips suitable for the test specimens (Figure 5) and the load was applied with cross head speed of 1mm/min. The specimens were tested with a full scale load of 50kg.

The tensile strength was calculated by the following formula:

$T.S = F (N)/A (mm)^2$

T.S: tensile strength (N/mm²),

Where

[F: peak load (Newton), A: cross sectional area (mm²)][4]

3. Surface roughness test:

A digital profilometer device (Surface roughness tester, TR200)(Figure 6) it was used to measure the surface roughness of the specimens. After deflasking, all the specimens were left unpolished. Each specimen was placed on a fixed and stable base and the device was placed in a way so that the stylus just touched the surface of the specimen. The stylus was then moved along the specimen surface for 10 mm length to generate the reading for that specimen. The surface roughness test was measured by a micrometer (Mm). Statistical analysis was conducted with descriptive statistics, independent sample test, one-way (ANOVA) and multiple comparison tests utilizing the least significant difference test (LSD).

Figure (3) Autoclave

Figure (4) Instron testing machine for transverse strength test

Figure (5) Instron testing machine for tensile strength test

Figure (6) Digital profilometer device for surface roughness test

RESULTS

The mean values of transverse strength and standard deviation for each studied group are presented in table (1).One-way ANOVA test showed no significant difference (P>0.05) between the means of the samples of all the test groups; autoclave processing slow, autoclave processing fast, and water bath group (Table 2).

The means and standard deviations for tensile strength for the samples of the experimental groups are presented in table (3). The mean difference between the different groups was insignificant (P>0.05) (Table 4).

The mean values and standard deviations for the experimental groups of surface roughness test are presented in table (5). One-way ANOVA test revealed that there was a highly significant difference (P<0.01) between the means of the test groups, as shown in Table (6). The samples processed in the autoclave with the short cycle had the highest readings (3.0622 Mm) for the surface roughness and were highly significantly different (P<0.01) compared with those of the water bath processing cycle, which had the lowest readings (1.8716 Mm). The mean of the surface roughness readings for the samples processed with the long autoclave cycle were in-between (2.722 Mm) and were significantly different (P<0.05) when compared with that of the water bath processing cycle. On the other hand, both the autoclave processing cycles were insignificantly different (P>0.05) when their means were compared. The (LSD) comparison among the groups is shown in Table (7).

DISCUSSION

According to the results obtained from this study and in regard to the experimental group and control groups of cross linking acrylic related to transverse strength test and tensile strength test, there was a non-significant difference between the autoclave of the two cycles (AL and AS) and water-bath (W) curing methods; this may be related to fact that this type of acrylic denture base material made in high degree of homogeneity that leads to absence of any changes occurred within this material. The type of bonds among the molecules of this type of acrylic resin is also more stable under higher temperatures.[30]Also the type of acrylic used in present study contains a cross linking agent which provides a sufficient number of bridges between linear macromolecules to form a three-dimensional network that increases the strength and rigidity of the resin.[29]

The results of this research showed no significant difference in transverse strength test between the means of the samples of all the test groups; autoclave processing long (AL), autoclave processing short (AS) and water bath (W) group. The results of this study were in agreement with Salwan[24], who found no-significant difference in transverse strength between autoclave and water-bath curing methods when using Vertex acrylic but he found there was a highly significant difference in transverse strength between autoclave and water bath processing methods when using high impact acrylic because this type of acrylic is modified by adding a rubber compound to improve strength properties.[7] The result of this study is not in agreement with Durkan et al[31], how studied the effect of autoclave polymerization on the transverse strength of high impact denture base polymers, the results revealed that polymerization in an autoclave led to a statistically significant increase in transverse strength for the materials evaluated when compared to the water bath. These conflicting results are due to using another curing cycle of autoclave processing. First they used autoclave-cured for 60ºC/ 30 min followed by130ºC/10 min, then they used autoclave-cured for 60ºC/ 30 min followed by130ºC/20 min

The tensile strength test is an important test which determines the resistance of the material to tensile or stretching force.[32]. Materials having a combination of reasonable tensile strength and elongation will be the tough material and those with low elongation will be brittle material.[4]

Furthermore, it is important to determine the surface roughness of the materials used for dental prostheses before their use in the mouth. Rougher surfaces can cause discoloration of the prosthesis, be a source of discomfort to patients and it may also contribute to microbial colonization and biofilm formation. Bacterial and fungal species have more of a propensity to adhere to rough denture base materials.[33,34] The surface roughness of a material used for removable prostheses is of importance because it affects, directly or indirectly; retention, staining resistance, plaque accumulation, oral tissue health, and patient comfort.[35,36]

On the other hand, the results of this study showed that polymerization in the autoclave led to a statistically significant difference in surface roughness test that indicate at this level of heat and pressure of autoclave processing cycle the beginning of reaction of the material to external elements.[37] These results was not in agreement with Manar et al[38], which revealed a non-significant difference between the water bath and the autoclave curing method may be due to using another processing cycle of autoclave 120 °C under 1.4 bar for 45 minutes and using another type of material (Vertex).

CONCLUSION

Our findings showed that there was no significant difference between the autoclave of the two cycles (long and short) and water-bath curing methods regarding transverse strength test and tensile strength test, but there was a significant difference between the results of surface roughness tests.

Regarding surface roughness tests, it was concluded there was a highly significant difference between the samples processed in the autoclave with the short cycle compared with the control group but there was a significant difference between the samples processed in the autoclave with the long cycle compared with the control group.

Table 1: descriptive statistics of transverse strength test (N/mm²)

Curing type	Mean	N	S.D.	Min	Max	S. E.
water bath	84.36800	10	10.819730	67.660	98.800	3.421499
autoclave long	89.07800	10	7.815524	82.630	101.770	2.471486
autoclave short	84.52800	10	8.212682	78.780	99.970	2.597078

Table 2:One-way ANOVA for transverse strength between test groups

transverse strength	Sum of Squares	d.f.	Mean Square	F-test	p-value
Between Groups	143.041	2	71.520	.874	.429
Within Groups	2210.374	27	81.866		
Total	2353.415	29			

Non-significant P>0.05

Table 3: descriptive statistics of tensile strength test (N/mm²)

Curingtype	Mean	N	S.D.	Minimum	Maximum	S.E.
water bath	31.05000	10	8.973789	20.000	44.080	2.837761
autoclave long	34.39000	10	4.254300	28.750	41.070	1.345328
autoclave short	33.12200	10	3.982402	28.100	37.650	1.259346

Table 4:One-way ANOVA for tensile strength between test groups

tensile strength	Sum of Squares	d.f.	Mean Square	F-test	p-value
Between Groups	56.855	2	28.428	.745	.484
Within Groups	1030.387	27	38.162		
Total	1087.243	29			

Non-significant P>0.05

Table 5: descriptive statistics of surface roughness test (Mm)

Curing type	Mean	N	S.D.	Minimum	Maximum	S.E.
water bath	1.87160	10	.397351	1.323	2.438	.125653
autoclave long	2.72200	10	.916296	1.615	3.793	.289758
autoclave short	3.06220	10	.711704	2.265	3.949	.225061
Total	2.55193	30	.852128	1.323	3.949	.155577

Table 6:One-way ANOVA for surface roughness between test groups

surface roughness	Sum of Squares	d.f.	Mean Square	F-test	p-value
Between Groups	7.521	2	3.761	7.501	.003**
Within Groups	13.536	27	.501		
Total	21.058	29			

Highly-Significant P<0.05**

Table 7: Least significant difference (LSD) between test groups

Curing type	Curing type	Mean Difference	S.E.	Sig.
water bath	autoclave long	-.850400*	.316650	.012*
	autoclave short	-1.190600*	.316650	.001**
autoclave long	autoclave short	-.340200	.316650	.292

P>0.05 Non-Significant, P*<0.05Significant, P<0.01 Highly Significant**

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