The Effect of Autoclave Processing on Some Properties of Cross-Linked Acrylic Denture Base Material

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Abstract:

Background: *Impact strength, surface hardness, water sorption and solubility are important properties for cross linking acrylic resin as denture base material so it is important to evaluate these properties after autoclave processing.*

Objectives: *The aim of this research is to investigate the effect of different time durations of autoclave processing on some physical and mechanical properties of cross linking acrylic denture base materials.*

Materials and methods: *Heat cured cross link acrylic was the acrylic denture base material included in this study. Total of (108) specimens were prepared. The specimens were grouped into: control group in which acrylic resin processed by conventional water bath processing technique and experimental groups in which acrylic resins processed by autoclave at 121°C, 210 KPa. The experimental groups were divided into group 1 (Fast) for 15 minutes. and group 2 (Slow) for 30minutes to study the effect of the autoclave processing, four tests were conducted: impact strength (Charpy tester), surface hardness (shore D), and water sorption and solubility test.*

Result: *The results were analyzed by ANOVA. There were no significant differences between the results of the processing techniques regarding impact, hardness, and water sorption and solubility tests.*

Conclusions: *The autoclave processing technique might also be a good alternative to the conventional water bath processing technique regardless to the cycle of autoclave processing either fast or slow.*

Key words: cross linked denture base acrylic resin, autoclave, and water bath.

Introduction:

The most commonly used material in construction of denture base since 1936 is polymethyl methacrylate [1]. Despite its popularity in satisfying aesthetic, simple processing and easy repair, it is still far from ideal in fulfilling the mechanical requirements of prosthesis [2]. Attempts to improve the mechanical properties of polymethyl methacrylate have taken the researcher through many avenues; cross-linking agents were added to the denture base materials and acrylic teeth by manufacturers to improve their physical and mechanical properties [3]; these agents are similar to methyl methacrylate chemically and structurally, therefore they will be incorporated into the growing polymer chain to provide a sufficient number of bridges or cross member between the linear macromolecules to form a three dimensional network-like structure that alter strength, decrease solubility and water sorption of the resin. Therefore ethylene-glycol dimethacrylate is added to the liquid to increase the organic solvent resistance, resistance to deformation, surface crazing, and fatigue and improve the physical properties of the set material [4, 5, 6].

The polymerization of PMMA is an additive reaction that requires activation of an initiator (benzoyl peroxide). This activation creates the first free radicals that start the polymerization chain reaction by opening the double bonds of the methyl methacrylate and the exothermic polymerization reaction has a tendency to increase rapidly as the temperature increases [7].

The water bath processing technique has been the most conventionally used polymerization technique. In spite of the advantages provided by this technique like the ease, simplicity and

cost-effectiveness, a major disadvantage has been the long processing time required [8].

The use of pressure cooker for denture polymerization was first reported by Muley in 1976 [9]; Previous studies of pressure cooker polymerization have shown comparable physical and mechanical properties to the water bath [8].The properties of processed heat-cured acrylic denture base in both a conventional water bath and an automatic pressure cooker were studied by Ming et al [10]; the results showed no significant difference in surface hardness of the processed acrylic denture base, but the pressure cooker had significantly shortened polymerization time. Another study was done to evaluate the effect of autoclave processing on some properties of conventional and high impact acrylic resin as denture base materials [11].

Material and methods:

Total of (108) specimens were prepared to be used in this study. They were divided into three main groups according to the type of processing used (water path and autoclave: short cycle and long cycle). Each group contained (12) specimens for each test (impact strength, surface hardness test and water sorption and solubility test).

Metal patterns (Fig 1) were constructed for hardness test with dimensions of (65mm x 10mm x 2.5mm) length, width and thickness respectively, and for impact strength test with dimensions of (80mm x 10mm x 4mm) length, width, and thickness respectively and for water sorption and solubility test with dimensions of 50 ± 1 mm in diameter and 0.5± 0.1 mm thickness

[12]. For water bath processing; the conventional flasking technique was followed in the mold preparation according to the required measurements of the adapted specimens. Each metal block was coated with petroleum jelly and immersed in the slurry stone (Type III hard stone, thixotropic, Zhermach/ Italy) which is prepared according to the manufacturer's instruction and poured into the lower half of the dental flask as in Fig 2. After setting of gypsum material, a layer of separating medium (Isodent, Spofadental/ Czech) was applied on the gypsum surface 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. 2ml of separating medium was applied by fine brush No. 0 onto the gypsum surface in each half of the flask then the mold was packed with cross-link acrylic resin dough (Sledgehammer, Keystone industries, USA) which was mixed according to the manufacturer's instruction (3:1) by volume and left under pressure 20 bar for 5 minute before clamping was done. The monomer liquid incorporate ethylene-glycol dimethacrylate 18-28%. Curing was carried out by placing the clamped flask in a water bath and processed by short curing cycle 90min at 74C° then temperature was increased to the boiling point 100°C for 30 minutes and all the tested specimens were conditioned in distilled water at 37C° for 48 hours before they were tested [12].

Autoclave curing was carried out by placing the clamped flask in a fully automatic autoclave (SW 22 plus sternweber, Italy) Fig 3 and processed by the preprogrammed cycles (Fast 121ºC/210KPa, 15 min). A fully automatic autoclave was filled, sterilized and exhausted at the touch of a button. Distilled water must be adjusted. Autoclave must be filled with distilled water until the water level (25mm) below the base of the Safety Valve Holder Min/Max lines

on the Reservoir Dip Stick. The clamped flask placed in the tray and pushed inside the chamber Fig3, then closed and secured start button and select standard programs (121ºC) for using as a curing cycle. In this cycle, the operation of autoclaves include air removal, steam admission and sterilization cycle (includes heating up, holding/exposure, and cooling stages) [13, 14]. The autoclave operated and started heating the water, then the temperature and pressure were raised till its reached (121ºC &210 KPa) respectively. When the temperature reached automatically at (121) temperature and pressure held automatically at (121 C &210KPa) respectively for 15 min., then automatically exhausted the steam the programmed cycle was finished. The metal flask was allowed to cool at room 30min., followed by complete cooling of the metal flask with 15 min. tap water. The acrylic patterns were removed from mold [4]; the same processing mentioned before repeated for long cycle group (121ºC &210 KPa) for 30 minutes [11].

Physical and mechanical tests utilized to examine properties:

Impact strength:

Impact strength test was conducted following the procedure given by the ISO 179 [15] with charpy type impact testing instrument (Fig 4). The specimen was supported horizontally at its ends and struck by a free swinging pendulum which released from a fixed height in the middle. A pendulum of 2 joules testing capacity was used. The scale reading gave the impact energy absorbed to fracture the specimen in joules when struck by a sudden blow. The

charpy impact strength of un-notched specimen was calculated in KJ/mm² as given by the following equation:

Impact strength = (E/ b.d) ×10³ [6] where

E: the impact absorbed energy in joules.
b: the width in millimeters of the test specimens.
d: the thickness in millimeters of the test specimens.

Surface hardness test:

Surface hardness was determined using (shore D) durometer hardness tester (TIME group Inc. company) according to American National standard Institute/American Dental Association (ANSI/ADA) No.12 [12] which is suitable for acrylic resin material (Fig 5). The instrument consists of blunt-pointed indenter 0.8mm in diameter that tapers to a cylinder 1.6mm. The indenter is attached to a digital scale that is graduated from 0 to 100 units. The usual method is to press down firmly and quickly on the indenter and record the maximum reading as the shore "D" hardness measurements were taken directly from the digital scale reading. Five readings with 1 cm apart between each two indentation along the specimen (the same selected area of each specimen), and an average of five readings was calculated.

Water sorption and solubility test:

The disks were dried in a desiccator containing freshly dried silica gel at 37ºC ± 2ºC for 24 hours with the use of an incubator Fig 6. Then removed to a similar desiccator at room temperature for one hour, then weighed. The cycle was repeated until constant

weight was attained, that is until the weight loss of each disk was not more than 0.5mg in every 24 hour period. The disks were then immersed in distilled water at 37ºC ± 1ºC for 7 days, after that time the disks were removed from water with tweezers, wiped with a clean dry hand towel, waved in the air for 15 seconds and weighed 1 minute after removal from the water.

The value for water sorption was calculated as follows:

$$\frac{W_2 - W_1}{S.A} = S$$

Where:

W2 = Weight after immersion (mg),
W1 = Conditioned weight (mg),
S.A = Surface area (cm^2),
S = Sorption (mg/cm^2)

The average of the determined values for the disks was recorded to the nearest 0.01 mg/cm^2.

To obtain the value of solubility test, the discs were reconditioned to a constant mass in the desiccator at 37 ºC ±2 ºC as done previously for sorption test and considered as the reconditioned mass.

The values for solubility were calculated for each disc from the following equation:

Solubility (mg/ cm^2) = condition mass (mg) - reconditioned mass (mg)/ Surface area (cm^2)

Result:

The mean of impact strength values and standard deviation for each studied groups are presented in Table 1.A one- way ANOVA test represent non-significant effect of autoclave processing either slow or fast on experimental groups (P>0.05) as shown in Table 2.The means and standard deviations of experimental groups presented in Table 3 belongs to hardness test. A one-way ANOVA yielded no significant differences between groups F (2, 27) = 0.339 in regard to hardness processed by autoclave in two cycles (slow and fast) as shown in Table 4.

The mean water sorption values and standard deviations for each experimental are presented in Table 5. A one way analysis of variance showed that the effect of curing type on water sorption was insignificant, F (2, 27) = 2.071, p =0.146 as shown in Table 6. The mean water solubility values and standard deviations for each study group are presented in Table 7. A one way analysis of variance showed that the effect of curing type on water solubility was insignificant, F (2, 27) = 1.839, p =0.178 as shown in Table 8.

Discussion:

The most conventional polymerized technique for acrylic resin processing is water bath; but in spite of the advantages provided by this technique like the ease, simplicity and cost-effectiveness, a major disadvantage has been the long processing time required [8]. Different polymerization methods have been used to improve the physical and mechanical properties of resin materials like: heat, light, chemical and microwave energy [16]. So the aim of this research is to study the effect of autoclave

processing technique (two different cycles short or fast cycle and long or slow cycle) on properties of cross linked acrylic denture base material.

According to the result obtained from this study to all experimental groups of cross linked acrylic related to (impact strength test, surface hardness test, and water sorption and solubility test), there was a non-significant difference between autoclave and water-bath methods, this may be related to fact that cross-linking made the material hard, rigid and having a greater effective molecular weight to withstand higher processing temperature [17, 18, 19, 20]. For cross-linked systems, however, chains are linked chemically; consequently, chains will not flow freely even under the application of heat and pressure [4, 20].

It may be that the temperature at 121°C was at or below the reversible melting transition and not enough to make an irreversible exothermic change. This led to the chemical and thermal stability of the material [18].

Cross-linking results in the formation of a network structure of covalently bonded atoms; primary linkages occur between chains, and the polymer actually becomes a single giant macromolecule and the spatial structure that allows chain sliding upon heating is not present in cross-linked materials [21]. So crosslinking improves mechanical, chemical and thermal properties. Melt flow index of crosslinked polymer should be lower because the crosslinking holds the material together even at the crystalline melting point of the uncrosslinked phases [19].

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Table 1 Descriptive statistics of Impact strength test (KJ /mm²)

Curing type	Mean	N	Std. Deviation	Minimum	Maximum	Std. Error
water path	7.70200	12	.586341	6.890	8.530	.185417
autoclave long	8.12800	12	.901170	6.870	9.270	.284975
autoclave short	8.53600	12	.811462	7.440	9.290	.256607

Table 2 ANOVA

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	3.478	2	1.739	2.876	.074*
Within Groups	16.329	27	.605		
Total	19.808	29			

*** Non significant, P>0.05**

Table 3 Descriptive statistics of surface Hardness test.

Curing type	Mean	N	Std. Deviation	Minimum	Maximum	Std. Error
water path	84.50000	12	1.313181	82.900	86.500	.415264
autoclave long	85.22000	12	2.955522	80.600	89.000	.934618
autoclave short	84.62000	12	1.641679	82.900	87.300	.519145

Table 4 ANOVA

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	2.976	2	1.488	.339	.715*
Within Groups	118.392	27	4.385		
Total	121.368	29			

Non significant, P>0,05*

Table 5: Descriptive statistics for water sorption (mg/cm²)

curing type	Mean	N	Std. Deviation	Minimum	Maximum	Std. Error
water path	.02920200	10	.002000615	.026390	.031700	.000632650
autoclave long	.03306480	10	.010124045	.025780	.051864	.003201504
autoclave short	.02763200	10	.002598644	.024500	.031820	.000821763
Total	.02996627	30	.006366950	.024500	.051864	.001162441

Table 6: ANOVA

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	.000	2	.000	2.071	.146*
Within Groups	.001	27	.000		
Total	.001	29			

Non significant, P>0,05

Table 7: Descriptive statistics for water solubility (mg/cm²)

curing type	Mean	N	Std. Deviation	Minimum	Maximum	Std. Error
water path	.00208000	10	.004298010	-.004000-	.008700	.001359150
autoclave long	-.00126000-	10	.005206236	-.008900-	.006700	.001646356
autoclave short	-.00052000-	10	.002151382	-.003100-	.002600	.000680327
Total	.00010000	30	.004207547	-.008900-	.008700	.000768189

Table 8: ANOVA

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	.000	2	.000	1.839	.178*
Within Groups	.000	27	.000		
Total	.001	29			

Non-significant, P>0, 05

Fig 1 Metal patterns

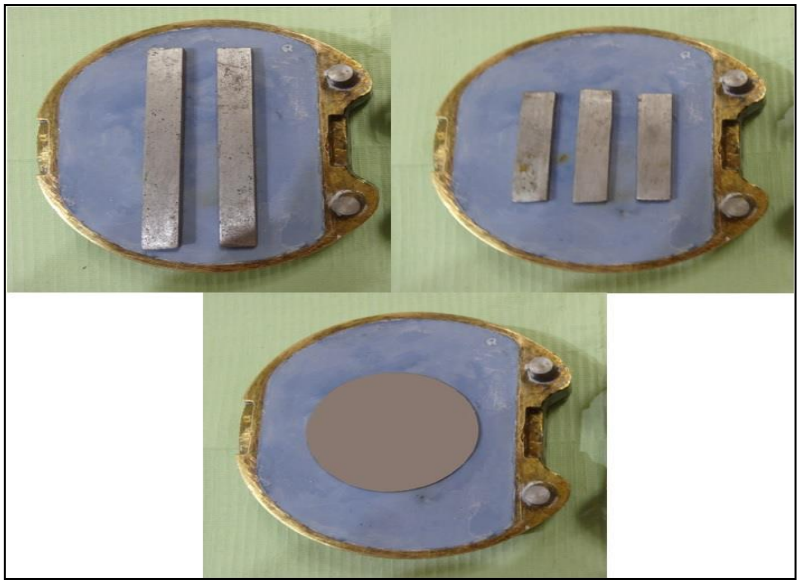

Fig 2 Metal pattern with flask

Fig 3 Autoclave

Fig 4 impact testing instrument

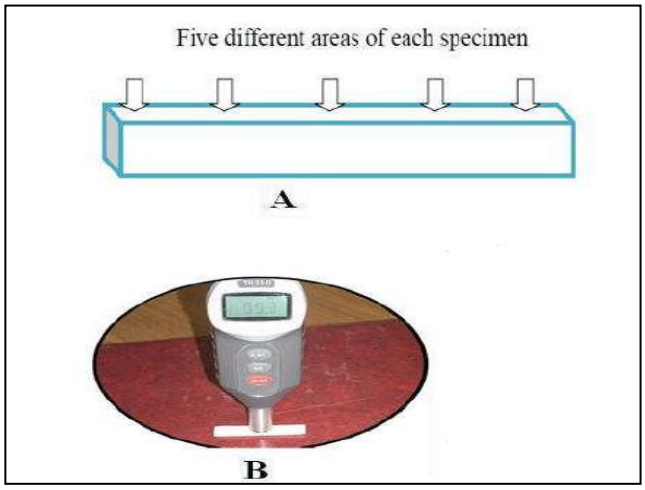

Fig 5 Hardness specimen and durometer hardness tester

Fig 6 Desiccator

تاثير المعالجة بالموصدة على بعض خصائص الاكريليك المتشابك المستخدم كقاعدة لطقم الأسنان

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المستخلص

قوة الصدمة، وصلادة السطح، وامتصاص الماء والذوبان هي من الخصائص الهامة للاكريليك المتشابك كمادة قاعدة طقم الأسنان لذلك من المهم تقييم هذه الخصائص بعد المعالجة بالموصدة.

الهدف من هذا البحث: هو دراسة تأثير فترات زمنية مختلفة من معالجة المُوصَدة على بعض الخصائص الميكانيكية والفيزيائية للاكريليك المتشابك كمادة قاعدة طقم الأسنان.

المواد والطرق: تم استعمال الاكريليك المتشابك المعالج حراريا في هذه الدراسة. المجموع الكلي للعينات التي تم تحضيرها هو 108. تم تقسيمها الى : مجموعة التحكم وفيها تم معالجة الاكريليك بحوض الماء التقليدي ومجموعات تجريبية تم فيها معالجة الاكريليك بالموصدة عند درجة حرارة 121° وضغط 210 كيلوباسكال. تم تقسيم المجموعات التجريبية الى المجموعة الاولى (المعالجة السريعة) لمدة 15 دقيقة

والمجموعة الثانية (المعالجة البطيئة) لمدة 30 دقيقة لدراسة تأثير معالجة الموصدة. أجريت أربعة اختبارات: قوة الصدمة، والصلادة السطحية واختبارامتصاص الماء واختبار الذوبان.

النتائج: عدم وجود فروق ذات دلالة إحصائية بين نتائج انواع المعالجة فيما يتعلق بقوة الصدمة، وصلادة السطح، وامتصاص الماء والذوبان.

الاستنتاجات: المعالجة بالموصدة قد تكون أيضا بديلا جيدا للمعالجة بحوض الماء التقليدي بغض النظر عن مدة المعالجة سريعة كانت أو بطيئة.

الكلمات الرئيسية: الاكريليك المتشابك كمادة قاعدة طقم الأسنان ، الموصدة، حوض الماء.