



## **The Effect of Autoclave on the Powder of (PMMA) on the Water Sorption, Solubility and Porosity**

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### **ABSTRACT**

To evaluate the influence of curing methods and temperatures as variables of water sorption, water solubility and present porosity in poly methyl methacrylate (PMMA) denture bases. The effect of autoclave processing on these properties has not been fully determined. The effect of autoclave on acrylic powder has not studied before. Vertex was the heat – cured acrylic denture base material included in this study. A total of 120 specimens were prepared, the specimens were grouped into three control groups of Vertex acrylic resin which (group A) as curing by conventional water- bath technique (74C° for 90 minutes then boil for 30 minutes), (group B) which curing by water- bath (100C° for 30 minutes), and (group C) which curing by autoclave (120 °C under 1.4 bar for 45 minutes). The other three groups of Vertex acrylic resin after treatment of powder by autoclave in 132 °C for 4 hours, and then grouped into (A<sub>1</sub>, B<sub>1</sub> and C<sub>1</sub>) which curing as previously. To study the effect of treatment of powder of acrylic resin by autoclave and the effect of autoclave processing. Three tests were conducted, water sorption and solubility tests, and the mean percent porosity (Electronic sensitive balance sensitive to 0.0001 g). The results were analysed to Descriptive Statistics and ANOVA test. There was a significant difference in water sorption test results in all studying groups. There was a non-significant difference in water solubility test results between studying groups. There was non-significant difference in present porosity test results between studying groups. The treatment of powder of acrylic resin by autoclave had a significant difference on water sorption, but had a non-significant difference on water solubility and percent porosity test in all test results. The autoclave processing technique might also be a good alternative to the conventional water- bath processing technique.

**Keywords:** Autoclave, polymerization methods, water sorption, porosity.

### **INTRODUCTION**

Many kinds of materials have been tried for artificial dentures from time to time in the quest for accurate, stable and lifelike dentures. Polymers meth- acrylic esters are the most commonly used materials for base of denture because of is excellent aesthetic values, adequate strength, simple manipulation and low cost.

An acceptable acrylic polymerization method is the one capable of the best properties of acrylic resin like transverse strength, monomer release and least porosity

(Bottega DM et al., 2004). Many attempts have been made to modify (PMMA) taking advantages of the broad scope of modification available in polymer chemistry. Polymerization by microwave or by autoclave has shown comparable physical and mechanical properties to the water bath technique. Curing process have been modified in order to improve the physical and mechanical properties of those materials and also to afford technical work of the professional (Azzari MJ et al., 2003).

Different polymerization methods have been used: heat, light and microwave energy (Grunewald AH et al., 1982). Water sorption has been recognized as a problem in the dimensional stability of acrylic dentures.

All polymers absorb water when immersed in water or when stored in high humidity. Some water molecules are adsorbed on the surface of a solid polymer while others are absorbed into the material, penetrating into structure of the solid polymer. This absorption, which derives from the polar properties of resin macromolecules through a diffusion process, increasing the capacity of the denture resin base to adapt to and be retained on the edentulous ridge tissue (Labella RS et al., 1990). The phenomena of absorption and adsorption are called sorption. Sorption results an increase in weight and swelling which might affect the polymer properties.

The high water up take is not suitable for the use in the dental materials. The rate of sorption is relatively high in the first few minutes and hours. As the concentration of water increases, the water sorption reduce and the sorption takes several days or even weeks to complete (Braden M, 1964). The change in the weight of polymer due to immersion in water may reduction in hardness and increase in flexibility due to the plasticizing effect of the water molecules in the polymer structure. Under environmental condition, the diffusion requires a period of 30 days to occur totally in acrylic resins (Barbosa CM et al., 2001). In a denture base material, water absorbed act as a plasticizer and affects the dimensional stability, subjecting the material to the internal stresses and possible crack formation (Tuna SH et al., 2008). Porosity of denture base resin continues to be one of the characteristics of (PMMA) which due to a variety of factors including, air entrapped during mixing, monomer contraction during polymerization, monomer vaporization associated with exothermic reaction, and the presence of residual monomer (Wolfaardt JF et al., 1986). Lack of porosity is essential for retaining a smooth, clean polishable surface. The polymerization cycle and denture base thickness both have an influence on porosity (Yannikakis S et al., 2002).

The aim of study was to investigate the effect of autoclave on some physical properties (PMMA) powder by curing process two cycles of water bath, and curing by autoclave.

## MATERIALS AND METHODS

The control groups of conventional heat curing denture base material of Vertex Regular Type 1 E-0120, Lot XU 034 LO5 Vertex –Dental by J.V oldenbaneveltin, 62 3705 HJ Zeist The Netherlands. These control groups were cured by water bath, type Derotor, Multi cure, Italy. Also

the control groups were cured by autoclave type Cristofoli, Biosseguranca, Vitale 21. The modification groups of (PMMA) which are modify the powder of acrylic by autoclave type- HIRAYAMA – HICTVE – HUA-110. Pr.unit 0.1 MPa – 1bar, 1.02Kgf/cm<sup>2</sup> = 14.5 Psi. The heat applied is 132°C and pressure for 4hr. This powder also curing by two different techniques (water bath in two cycles and by using autoclave). The two cycles of water bath are: - The first cycle was done by Curing in water bath at 74°C for 90 minutes. Then raise the temperature to 100°C for 30 minutes. The second was done by curing in water bath at 100°C for 30 minute (Abood NL, 2007). The curing by autoclave at 120°C fewer than 1.4 bars for 45 minutes (Abdulwahab SS and Alnakkash WAH, 2012).

In this vitro study a total of 180 specimens were prepared, the specimens, water bath and by autoclave, so these groups were: Group A, Group B and Group C, also modification groups processed by both two cycles of water bath and by autoclave, so these groups were: Group A<sub>1</sub>, Group B<sub>1</sub> and Group C<sub>1</sub>.

## Water sorption and solubility tests

To measure the water sorption and solubility, 60 samples prepared with dimensions (10<sub>x</sub>12<sub>x</sub>4) mm, 10 samples from each group. The samples were dried in air at 37°C until their weight was constant, and maintained at 37°C for one week. After this time, the samples were immersed in water and removed, blotted to remove surface water, dried in air for 15seconds, this result was recorded as (**m**<sub>1</sub>). The samples were then immersed in distilled water and weight, this result was recorded as (**m**<sub>2</sub>). Samples were placed in the desiccators that contained anhydrous calcium and dried until the final constant mass was recorded (**m**<sub>3</sub>). The volume of samples (**V**) was calculated by multiplying (length<sub>x</sub> width<sub>x</sub> thickness).

To calculate water sorption (**Wsp**) and solubility (**Wsl**) the following equations (1) and (2) were used (Podgoski M, 2010).

$$\mathbf{Wsp} = \mathbf{m}_2 - \mathbf{m}_1 / \mathbf{V} \quad (1)$$

$$\mathbf{Wsl} = \mathbf{m}_2 - \mathbf{m}_3 / \mathbf{V} \quad (2)$$

## Porosity test

To measure porosity, the classical sorption method was used in this study, 60 samples were prepared, and 10 samples from each group with dimensions (10<sub>x</sub>12<sub>x</sub>4) mm, the samples were dried in a desiccator containing silica gel under a vacuum. They were weighted daily by an analytical balance capable of measuring to 0.0001 g until a constant weight was reached. With samples dried, two weights were made, one with samples in air and other with the samples immediately immersed in distilled water, and then the samples were weighted. There were then

**Table1.** Descriptive statistics of water sorption test results.

	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
A	10	6.55377	1.293584	.409067	4.167	8.333
A <sub>1</sub>	10	6.00690	1.866831	.590344	4.166	8.333
B	10	6.64440	1.622094	.512951	3.788	8.333
B <sub>1</sub>	10	5.12370	2.370498	.749617	1.894	9.470
C	10	5.08570	1.805245	.570869	2.083	8.333
C <sub>1</sub>	10	3.68670	1.049099	.331754	2.071	5.681

**Table2.** F – test by ANOVA table of water sorption results

	Sum of Squares	df	Mean Square	F	P-value
Between Groups	62.766	5	12.553	4.239	.0030
Within Groups	159.916	54	2.961		
Total	222.682	59			

weighed at regular intervals until a constant mass was reached indicating a state of water saturation for a period of 30 days.

The samples were removed from the water and excess water was removed by blotting with filter paper, and again the samples were weighed, one in air and the other with the samples immediately immersed in distilled water. The porosity calculations were made using the following equations (Keller JC and Lautenschlager EP, 1985; Oliveira VMB et al., 2003).

$$V_d = \frac{m_d - m_d'}{\rho_w} \quad (1)$$

$$V_s = \frac{m_s - m_s'}{\rho_w} \quad (2)$$

$$\% \text{ Porosity} = \left( \frac{V_s - V_d}{V_d} \right) \times 100 \quad (3)$$

Where  $V_d$  = dried specimen volume;  $m_d$  = mass of dried specimen in air ;  $m_d'$  = mass of dried specimen in water,  $\rho_w$  = density of water,  $V_s$  = volume of specimen saturated by water,  $m_s$  = mass of saturated specimen in air; and  $m_s'$  = mass of saturated specimen in water.

In equation (1) and (2), the volumes were determined using  $\rho_w = 1000 \text{ Kg/m}^3$ , the porosity could be calculated by the volume of saturated specimen minus the dried specimen volume divided into dried specimen and multiplied by 100 total percent of porosity for each specimen in equation (3). The porosity calculation was based on mass and volume of each sample before and after immersion in water, and the density of water (Compagnoni MA et al., 2004).

## RESULTS

### Water sorption and solubility test

Descriptive statistical analysis revealed a significant difference between all groups of Vertex in both control

groups (A, B and C), and the modified groups ( A<sub>1</sub>, B<sub>1</sub>, C<sub>1</sub>).

The minimum value of water sorption was in the group( C<sub>1</sub>) which equal to 3.6867 mg / cm<sup>3</sup> while the maximum value was in the group( B )which equal to 6.6444 mg/ cm<sup>3</sup> as were shown in (Tables1 and 2 ).

Descriptive statistical analysis revealed a non – significant difference between all groups of vertex in both control types (A, B and C), also the modified groups of Vertex (A<sub>1</sub>, B<sub>1</sub> and C<sub>1</sub>) in water solubility test.

The minimum value of water solubility was in group C<sub>1</sub> which equal to 4.466 mg / cm<sup>3</sup> while the maximum value was in the group C which equal to 5.7214 mg/cm<sup>3</sup> as were shown in (Tables3 and 4).

### Porosity test

Descriptive statistical analysis revealed a non-significant difference between all groups of Vertex in both control types (A, B and C), also the modified groups of Vertex (A<sub>1</sub>, B<sub>1</sub> and C<sub>1</sub>) in porosity test. The minimum value of porosity was in group (C<sub>1</sub>) which equal to 0.69715% while the maximum value was in group( A ) which equal to 1.02079% as were shown in (Tables5 and 6).

## DISCUSSION

The term autoclave is also used in processing materials by elevated temperature and pressure. The effect of the autoclave on the acrylic powder itself has not been evaluated before. There is a continuing effort to improve the properties of denture base materials. Curing

**Table3.** Descriptive statistics of water solubility test results

	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
A	10	5.49290	1.882373	.595259	2.270	7.905
A <sub>1</sub>	10	5.71210	2.685710	.849296	2.165	9.901
B	10	5.45650	1.202428	.380241	3.925	7.869
B <sub>1</sub>	10	5.25990	1.559847	.493267	3.788	7.692
C	10	5.72140	1.476830	.467015	3.483	7.575
C <sub>1</sub>	10	4.46600	2.155495	.681627	1.894	9.058

**Table4.** F- by ANOVA table of water solubility results

	Sum of Squares	df	Mean Square	F	P-value
Between Groups	10.904	5	2.181	.6100	.6930
Within Groups	193.163	54	3.577		
Total	204.066	59			

**Table5.** Descriptive statistics of porosity test results

	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
A	10	1.020790	.4422391	.1398483	.31580	1.6670
A <sub>1</sub>	10	.839650	.2051769	.0648826	.46670	1.1818
B	10	.990570	.3158792	.0998898	.53330	1.5466
B <sub>1</sub>	10	.897870	.2451328	.0775178	.53850	1.3333
C	10	.876950	.4013481	.1269174	.42820	1.6250
C <sub>1</sub>	10	.697150	.2510358	.0793845	.41110	1.2677

**Table6.** F- by ANOVA table of porosity results

	Sum of Squares	df	Mean Square	F	P-value
Between Groups	.671	5	.1340	1.296	.2790
Within Groups	5.595	54	.1040		
Total	6.266	59			

processes have been modified in order to improve the physical and mechanical properties of those materials.

Reduction of water sorption and water solubility as well as improvement of biocompatibility is very important because of still is a major cause of clinical failure of denture base material.

Porosity can weaken a denture base, promote staining, render denture anaesthetic due to colour change, harbour organisms such as *Candida albicans*, and can cause bond failures between artificial tooth and denture base resin (Pero AC et al., 2010). A porous

denture base is very difficult if not impossible to finish and polish and thus unwanted of aspect of denture fabrication.

Water sorption in the acrylic resin, partly may be due to the presence of deeper porosity and surface flows, the maximum value of water sorption in group B in this study may be in the presence of voids or pores which lead to diffusion of ionic molecules of water between the polar bonds of acrylic resin. Immersion of the polymer in water also results in certain components of polymer dissolved in medium. The net result of sorption is increase in weight

due to absorption of water and also the loss of weight because of dissolution of certain components from polymer into water.

Water solubility may be due to decrease the potential sites of water exchange to occur and can be related to the leach of soluble materials like residual monomer and plasticizers. In this study there were no significant statistical differences between all control and modified groups. This may be due to the small amount of residual monomer released.

An adequate solution for storing specimens must be used to measure porosity by water absorption. Porosity occurs due to the air trapped during mixing, monomer contraction and evaporation of monomer during curing (Vshma B and Nandish BT, 2006).

There were no significant statistical differences in mean percent porosity between the acrylic specimens cured by heating in water-bath with two cycles, and cured by heating in autoclave with elevated temperature and pressure. The minimum value of mean percent porosity was in group( C<sub>1</sub> ) which was equal to 0.69715 while the maximum value of mean percent porosity was in group( A )which was equal to 1.02079. The mean percent porosity may be related to the absolute density of the acrylic resin and the weight of the specimen, pressure in the initial stages of polymerization was considered to minimize the mean percent porosity (Pero AC et al., 2011).

## CONCLUSION

Within the limitation in this my vitro study, concluded that: There was a significant statistical difference in mean value of water sorption between all groups; reduced value was seen in group (C1). Also There was a non-significant difference in mean value of water solubility between all groups. Another concluded, there was a non-significant difference in mean percent porosity between all groups, and last the treatment of acrylic powder with autoclave decrease water sorption and solubility and percent porosity in all groups.

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