



## **The effect of polypropylene fibers in different lengths on some properties of heat-cured acrylic resin processed by autoclave.**

**Dr. Husam Mejbel Khalaf, B.D.S, M.Sc .\***

### **Abstract**

**Background:** some properties of heat-cured acrylic resin are required to be improved (like flexural strength, hardness and porosity) by adding different types of fibers like polypropylene fiber, also needed to determine these properties with addition of polypropylene fibers and curing the polymer by autoclave. The aim of this study was to evaluate the effect of adding polypropylene fibers in two different lengths (6mm, 12mm) and concentration 2%, and the effect of autoclave processing on some properties (flexural, surface hardness and porosity).

**Material and method:** A total No. of 120 specimens were prepared in this study and divided into two main groups according to length of fibers (6mm, 12mm), each main group was subdivided into two groups according to method of adding the fiber (directly to the powder or immersed in monomer). Each group of them contains 10 specimens for each test.

**Result:** in this study, the transverse strength and surface hardness of heat-cured acrylic resin reinforced by randomly oriented polypropylene fibers (12mm, 2%) regardless of application method with higher significance than other groups. Fiber reinforcement and autoclave processing showed non-significant effect on porosity of heat-cured acrylic resin according to ANOVA t-test.

**Conclusion:** Reinforcement by randomly oriented polypropylene fiber (12mm, 2%) added with and without immersion in monomer is better than other groups according to transverse strength and surface hardness with autoclave processing but there is no effect of added fibers on porosity test.

**Key words:** acrylic resin, polypropylene fibers, autoclave.

### **Introduction**

One of the most widely used materials in prosthetic dentistry is polymethyl methacrylate (PMMA). Excellent appearance, ease in processing, and reparability, make PMMA an excellent denture base material. However, the primary problem is its poor strength characteristics, including low impact and flexural strength<sup>[1-4]</sup>.

Strengthening the acrylic resin prosthesis can be approached by modifying or reinforcing the resin. One of the most common reinforcing techniques is the use of different types of fibers. The addition of fibers to acrylic resin has the potential to improve the mechanical properties of the material. Effective fiber reinforcement is dependent on many

---

\* College of dentistry, university of Al-mustansiria

variables including the material used, the percentage of fibers in the matrix and their modulus and distribution, fiber length, fiber orientation and fiber form<sup>(5)</sup>. Over the years, various types of fibers such as carbon<sup>(6)</sup>, aramid<sup>(7)</sup>, polyethylene<sup>(8)</sup>, and glass<sup>(9)</sup>. In this study we added polypropylene fibers<sup>(10)</sup>. Hardness is a simple and effective way to assess the degree of conversion of dental polymers<sup>(11)</sup>, and the degree of conversion of conventional heat-polymerized and self-curing acrylic resins<sup>(12)</sup>. Porosity is a property of solids that relates to their structure and is expressed in the presence of voids (pores) between separate grains, layers, crystals, and other elements of a coarse structure of a solid<sup>(13)</sup>. The generation of porosity in PMMA denture base resin is apparently a complex phenomenon with a multifactorial origin<sup>(14)</sup>. The transverse (flexural) strength is a combination of compressive, tensile, and shear strengths, all of which directly reflect the stiffness and resistance of a material to fracture<sup>(15)</sup>. This property of acrylic resins depends on several factors, such as polymer molecular weight, polymer bead size, residual monomer level, plasticizer composition, amounts of cross-linking agents, internal porosity of the polymer matrix, denture base thickness, patient factors, type of polishing, and action of chemical agents<sup>(16)</sup>. Hardness is a measure of the resistance to plastic deformation and is measured as a force per unit area of indentation. Hardness influences ease of cutting, finishing, and polishing an object and its resistance to in-service scratching<sup>(17)</sup>. Most of the methods for measuring hardness consist of making an indentation in the surface of a material with a specified force in a controlled and reproducible manner and measuring the size of the indentation<sup>(18)</sup>.

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<sup>(19)</sup>. The use of pressure cooker for denture polymerization was first reported by Muley in 1976<sup>(20)</sup>.

Indian researchers like Muley, Sidhaye A.B, and Undurwade J.H extensively investigated the pressure cooker polymerization technique. Researchers studied the polymerization of denture base material in a domestic pressure cooker<sup>(21)</sup>. Conventional acrylic resin material can be used for this technique and requires less than 1 hour for polymerization and utilizes conventional equipment. Previous studies of pressure cooker polymerization have shown comparable physical and mechanical properties to the water bath technique.

## Material and Methods

A total of 120 specimens were prepared to be used in this study. They were divided into two main groups according to length of polypropylene fibers (6mm, 12mm). Each main group was subdivided into two subdivisions according to the method of addition (with powder or immersed in monomer for 10 minutes in a Petri dish for better bonding of fibers with PMMA resin)<sup>(22)</sup>. After the fibers were removed from the monomer, excess liquid was allowed to dry and fibers were mixed thoroughly with the polymer powder; each group of them contain 10 specimens for each test.

Preparation of heat-cured specimens for transverse strength, surface hardness and porosity:

Metal patterns were constructed with the dimensions of (65mm x 10mm

x 2.5mm) length, width and thickness respectively for all specimens (transverse strength, surface hardness and porosity) <sup>(23)</sup>. The conventional flasking technique was followed in the mould preparation according to the required measurements of the adopted specimens. Each metal block was coated with petroleum jelly and immersed in the slurry stone (Type III hard stone, thixotropic, Zhermack/Italy) which is prepared according to the manufacturer's instruction and poured into the lower half of the dental flask as in Fig. 1. After setting of gypsum material, a layer of separating medium 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 cold mold seal (P.D. Pink color 1b separating film/ Switzerland) was applied by fine brush No. 0 onto the gypsum surface in each half of the flask then the mold was packed with acrylic resin dough (Vertex/J.v.Oldenbamevetin Zeist, Netherlands.) which was mixed according to the manufacturer's instruction (3:1) by volume and left under pressure 20 bar for 5 minutes before clamping was done. Curing was carried out by placing the clamped flask in a fully automatic autoclave (SW 22 plus sternweber, Italy) Fig. 2 and processed by the preprogrammed cycles (Fast 121°C/210KPa, 15 min). A fully automatic autoclave 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 Fig. 3, then closed and secured start button and select standard

programs (121°C) 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) <sup>(24)</sup>. The autoclave operated and started heating the water, then the temperature and pressure were raised till its reached (121°C & 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 30 min., followed by complete cooling of the metal flask with 15 min. before deflasking. The acrylic patterns were removed from mould <sup>(25)</sup>.

Physical and mechanical tests utilized to examine properties:

### 1. Transverse strength

The test was achieved by using instron testing machine, each specimen was positioned on bending fixture, consisting of 2 parallel supports 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 (Fig.4).

The transverse bend strength was calculated in N/mm<sup>2</sup> using the following formula: Transverse strength =  $3Pl/2bd^2$  (28)

All the specimens were immersed in distilled water at 37°C for 48h before being tested (ADA specification, No1999).

### 2. Surface hardness:

This test 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, 1999 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.

### 3. Porosity test:

The method used is according to ASTM standards <sup>(27)</sup>. At first the weight of specimens were measured after flasking (dry  $W_d$ ) by digital electronic balance then immersed in distilled water at 37 °C at vacuum to remove all air and replaced by water. After vacuum, we measure the weight of specimens (saturated with water  $W_s$ ) by electrical balance. at the last we measure their weight when the specimens immersed in water ( $W_i$ ) so the porosity was calculated from this equation.  $A.P = (W_s - W_d / W_s - W_i) \times 100\%$

## Results

The mean, standard deviation, standard error in addition to maximum and minimum of transverse strength of control and experimental groups shown in table (1); one way analysis of variance (ANOVA f-test) was used to examine the effect of polypropylene fiber (2%) by autoclave processing on the transverse strength of the specimens (there is significant difference between tests groups P-value 0.016) table2.

The LSD showed a significant difference between group C1 (12mm) without immersion in monomer) and

groups B1 and B2 (6mm without and with immersion in monomer respectively); the same result related to group C2 (12mm fibers immersed in monomer).

For porosity tests; mean, standard deviation, standard error, maximum and minimum shown in table3. ANOVA f-test of variance showed non-significant difference (P=0.061) between control group (A) and experimental groups (B1, B2, C1, C2) also within groups table 4.

The mean, standard deviation, standard error, maximum and minimum of surface hardness of control group (A) and experimental groups (B1,B2,C1,C2) in table5 and there is non-significant difference exist between groups according to ANOVA f-test (p=0,076) table 6.

According to LSD, the addition of polypropylene fiber to powder without immersion in monomer with 6mm length (B1) did not improve the hardness when compared with 6mm length (B2)  $t = 0.019$ . The same result when comparison occur between B1, C1 and C2 the value of t respectively (0.044 , 0,010) so for hardness the addition of polypropylene to powder with 6mm length and 2% did not improve hardness when compared to other groups (B2, C1 and C2).

## Discussion

Studies on the effect of autoclave polymerization on the transverse strength of denture base polymers revealed that polymerization in an autoclave lead to a statistically significant increase in transverse strength for some types of denture base polymers <sup>(28)</sup>. So the result agreed with Durkan et al 2008 <sup>(26)</sup>. ANOVA means of transverse strength showed significant improve in transverse strength between control group (A) and reinforced groups with polypropylene

fibers in 2% and two different lengths (6mm,12mm) and different applied methods (B1,B2,C1,C2), this result due to fact that related to the presence of fibers in resin thus ensuring transmission of loads from matrix to fiber, which also arrest the cracks leading to an increase in the strength of the resin by which allowing the resin to tolerate the load applied more than when the specimens had no fiber in their structure because in polymer-fiber composites, the fibers are embedded in a polymer matrix, which binds the fibers and forms a continuous phase surrounding the fibers, so the polymer matrix transfers loads to the fibers, which are the stronger component of the composite <sup>(29)</sup>. The connection between those two materials (fiber and polymer) plays an important role in load transmission from the matrix to the fiber material <sup>(30)</sup>. This could be due to good adhesion between the fibers and the matrix, enabling the transfer of a greater load from the matrix to the fibers.

In this study, shore (D) hardness tester was used which is suitable for measuring the hardness of acrylic resin <sup>(31)</sup>. Shore durometer type (D) hardness tester eliminate problem with elastic recovery owing to its use of a method that measures the depth of the loaded indentation under loading condition directly by screen which show its reading <sup>(31)</sup>. ANOVA test for means of surface hardness showed non-significant difference exist between control group and reinforced groups with polypropylene fibers (table 6). This result could be related to the fact that the surface hardness test is concerned with the outer surface and not with inner surface of the composite where these fibers were not near the surface of the composite. There was increase in surface hardness property associated with (12mm length and 2% concentration added directly to powder

) and this can give an indication that these fibers were located nearer to the outer surface of the mixture rather than the inner surface. This result agreed with Unalan and Dikbas, 2007 <sup>(31)</sup> and Nagham 2008 <sup>(32)</sup>.

Porosity in acrylic resin is a complex phenomenon of multifactorial origin. It appears to depend partly on the material/ polymerization method combination and the flasking technique used. Once acrylic resin has been polymerized and pores form in its mass, it is very difficult to determine whether the porosity resulted from the material itself, the polymerization cycle, or handling during the flasking procedure <sup>(33)</sup>. The lower value of porosity in slow curing cycle could be attributed to the role of the pressure accelerating the polymerization, the higher pressure is instantly transmitted to the resin dough and prevents the monomer from boiling <sup>(34)</sup>. Table 4 showed no effect to randomly oriented polypropylene fibers in two different lengths on porosity of vertex acrylic resin.

## References

- 1- Jagger DC, Harrison A, Jankdt KD. Review.The reinforcement of dentures. Oral Rehabil 1999;26:185-194.
- 2- Johnston EP, Nicholls JI, Smith DE. Flexure fatigue of 10 commonly use denture base resins. J Prosthet Dent 1981;46:478-83.
- 3- Darbar UR, Huggett R, Harrison A. Denture fracture--a survey. Br Dent J 1994;176:342-5.
- 4- M. Vojdani, AAR. Khaledi, Transverse strength of reinforced denture base with metal wire and E-glass fibers, J. Of Dentistry, Tehran university of Medical science, Tehran, Iran(2006; vol:3, No.4).
- 5- Kassab Bashi T, Al-Nema LM. Evaluation of Some Mechanical Properties of Reinforced Acrylic Resin Denture Base Material(An In Vitro Study). Al-Rafidain Dent J. 2009; 9(1):57-65.
- 6- Bowman AJ, Manley TR. The elimination of breakages in upper

- dentures by reinforcement with carbon fibers. *Br. Dent. J.* 1984; 156: 87–9.
- 7- Berrong JM, Weed RM, Young JM. Fracture resistance of Kevlar – reinforced polymethylmethacrylate resin a preliminary study. *Int. J. Prosthodont.* 1990; 3:391–5.
  - 8- Ladizesky NH, Pang MKM, Chow TW and Ward IM. Acrylic resin reinforced with woven highly drawn linear polyethylene fibers. Mechanical properties and further aspects of denture construction. *Aust. Dent. J.* 1993; 38: 28–38.
  - 9- Vallittu PK. The effect of glass fiber reinforcement on the fracture resistance of a provisional fixed partial denture restoration. *J. Prosthet. Dent.* 1998; 79: 125–30.
  - 10- Dr. Kumar Shalabh, Dr. Gupta Ajay, Dr. Tomar Leena (2010):” Comparison of Transverse Strength of Conventional Denture Base Resin and Reinforced Denture Base Resin with glass fibers, polyethylene and polypropylene fibers.” *J. Orofacial and health science*, vol. 1 and issue 2.
  - 11- Rueggeberg F.M. (2002):”From vulcanite to vinyl, a history of resins in restorative dentistry” *J. Prosth. Dent.*, 87:364-377.
  - 12- Lee HB. , Khang G., and Lee JH. (2003):- ”Polymeric Biomaterials: Principles and applications.” Park JB, Bronzino JD, CRC press. USA.
  - 13- Tager A. (1978):”Physical Chemistry of Polymers.” Moscow, MirPublisher, p: 59-627.
  - 14- 14.Wolfaardt J.F., Cleaton-Jones P., Fatti P. (1986):”The occurrence of porosity in a heat-cured poly (methyl methacrylate) denture base resin.” *J. Prosthet. Dent.*; 55:393-400.
  - 15- Jagger D.C., Jagger R.G., Allen S.M., Harrison A. (2002):”An investigation into the transverse and impact strength of high strength denture base acrylic resins.” *J. of Oral Rehabilitation*, Blackwell Science Ltd., 29; 263-267.
  - 16- Orsi I.A. and Andrade V.G. (2004):”Effect of chemical disinfectants on the transverse strength of heat-polymerized acrylic resins submitted to mechanical and chemical polishing.” *J. of Prosthetic Dent.*; 92: 382-8.
  - 17- Powers J. M., Sakaguchi R. L. (2006):”Craig’s Restorative dental materials.” 12th Ed. St. Louis Mosby Co. Ch.1p: 7, Ch.21 p:514-515,517,
  - 18- O’Brien W. J. (2002):”Dental material and their selection.” 3rd Ed. Quintessence Publishing Co, Inc. Ch.6 p: 135.
  - 19- Banerjee R, Banerjee S, Prabhudesai P.S., Bhide S.V.(2010):”Influence of the processing technique on the flexural fatigue strength of denture base resins: An in vitro investigation.” *Indian Dent Assoc.*, 21:3915.
  - 20- Sidhaye A.B. (1981):”Polymerization shrinkage of heat cured acrylic resins processed under steam pressure.” *Indian Dent Assoc.*, 53: 49-51.
  - 21- Undurwade J.H, Sidhaye A.B. (1989):”Curing acrylic resin in a domestic pressure cooker: A study of residual monomer content.” *Quintessence Int.*; 20(2): 123-129.
  - 22- John J, Gangadhar SA, Shah I. Flexural strength of heat-polymerized polymethyl methacrylate denture resin reinforced with glass, aramid, or nylon fibers. *J Prosthet Dent.* 2001;86:424–427.
  - 23- American Dental Association Specification No.12. Guide to dental materials and devices. 10th ed. Chicago; 1999. p.32.
  - 24- Judelson H.S. (2004):” Operation of the autoclaves.” An excellent overview of autoclave operation posted by Dr. Howard Judelson at University of California at Riverside.
  - 25- Craig R.G., Powers J.M (2002):”Restorative dental materials.” 11th Ed. St. Louis Mosby Co. ch.21, p: 636-689.
  - 26- Durkan R., Ozel M.B., Bagis B., and Usanmaz A. (2008):”In vitro, Comparison of autoclave polymerization on the transverse strength of denture base resins.” *Dental Materials J.*, 27(4): 640-642.
  - 27- C830, Annual Book of ASTM Standards, American Society for Testing and Materials, U.S.A., Vol.15.01,(1988).
  - 28- Anusavice K. J. (2007):” Phillips’s sciences of dental materials.” 11th Ed. Saunders Co. Philadelphia, p: 162-169.
  - 29- Vallittu P.K. (1996):-”A review of fiber-reinforced denture base resins.” *J. Pros. Dent.*; 5(4): 270-276.
  - 30- Herrera-Franco PJ. and Drzal LT. (1992):-”Comparison of methods for the measurement of fiber bond composites.” *Composites*; 23: 2-27.
  - 31- Unalan F. and Dikbas I. (2007):”Effects of mica and glass on surface hardness of acrylic tooth material.” *Dent. Material J.*, 26(4): 545-548.

- 32- Nagham B. (2008):-"Effect of addition of different lengths and concentrations of silane treated glass fibers on some properties of heat cured acrylic resin." A Master thesis, Department of prosthodontics, University of Baghdad.
- 33- Yannikakis S., Zissis A., Polyzois G., Andreopoulos (2002):" Evaluation of porosity in microwave-processed acrylic resin using a photographic method." J. Prosthet. Dent.; 87: 613-9.
34. Ming X.C., Changxi S., Weizhou H. (1996):"Rapid processing procedure for heat polymerization of poly (methyl methacrylate) in a pressure cooker with automatic controls." J. Prosthet Dent.; 76: 445-447.

Table 1- Descriptive of transverse strength test

	N	Minimum	Maximum	Mean	Std. E	Std. D
A	10	94.67	122.00	101.8350	4.11422	10.07774
B1	10	92.20	96.10	94.0783	0.58969	1.44443
B2	10	90.17	98.12	94.1750	1.04265	2.55397
C1	10	93.49	126.06	108.1333	4.70897	11.53458
C2	10	98.11	128.30	107.1633	4.53042	11.09721

Table 2-ANOVA test for the means of transverse strength of all groups

ANOVA-Table					
A & other groups	SS	Df	MS	F-test	P-value
Between groups	1104.17	4	276.04	3.767	0.016 S

There is a significant difference

Table 3- Descriptive of porosity test

	N	Minimum	Maximum	Mean	Std. E	Std. D
A	10	14.72	28.09	20.6480	2.44066	5.45749
B1	10	7.22	19.22	13.6420	2.59788	5.80905
B2	10	9.78	22.00	16.4960	2.10315	4.70278
C1	10	12.48	26.55	22.7840	2.64885	5.92302
C2	10	15.50	18.45	16.8500	0.48144	1.07652

Table 4- ANOVA test for the means of porosity of all groups

ANOVA-Table					
A & other groups	SS	Df	MS	F-test	P-value
Between groups	262.2	4	65.55	2.689	0.061 NS

There is a non-significant difference

Table 5- Descriptive of surface hardness test

	N	Minimum	Maximum	Mean	Std. E	Std. D
A	10	86.2	88.4	87.158	0.3323	0.8139
B1	10	82.2	87.3	85.833	0.8057	1.9735
B2	10	86.5	89.0	87.800	0.4274	1.0469
C1	10	86.1	89.0	87.500	0.5209	1.2759
C2	10	86.3	89.7	88.033	0.5806	1.4222

Table 6- ANOVA test for the means of hardness of all groups

ANOVA-Table					
A & other groups	SS	Df	MS	F-test	P-value
Between groups	17.95	4	4.489	2.413	0.076 NS

There is a non-significant difference



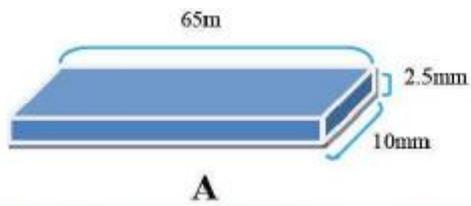
Fig. 1



Fig. 2



Fig.3



A



B



C

Fig.4

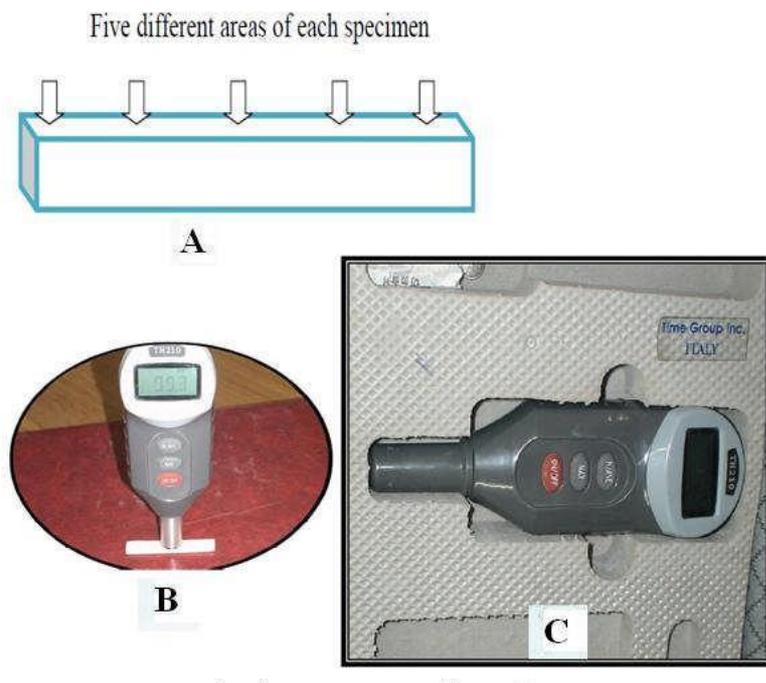


Fig.5