



PREPARATION OF METHYL METHACRYLATE –STYRENE COPOLYMER USING TO DENTURE PROTEASES

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Abstract: Poly methyl methacrylate (PMMA) is the most common material used in Prosthetic dentures [1-2]. Various attempts have been made to avoid breaking prosthesis are like changing the chemical structure of the resin with the addition of cross linking agents or copolymerization [3-5]. The innovative method for improving the physical properties of the prosthesis and increase strength, a copolymer of styrene - methyl methacrylate (St-Co-MMA) was proposed. The St-Co-MMA by weight percentages (20-80, 40-60, 50-50, 60-40 & 80-20) were synthesized. Increasing styrene $w_v\%$ in copolymer this causes that the tensile strength increased until it reached a certain value, at the maximum tensile strength. The tensile strength decreased while more than styrene copolymer. St-Co-MMA characterized by FTIR, NMR spectroscopic methods. TGA, decomposition temperature (T_D) and tensile strength were measured according to ISO 1567 standard. In this project its showed the highest tensile strength of the synthetic styrene - methyl methacrylate copolymer was (20-80) $w_v\%$.

Keywords: Poly methyl methacrylate (PMMA), Styrene - Methyl methacrylate Copolymer

Introduction: Until about 60 years ago, despite rapid advances in technology, polymers, polymethyl methacrylate (PMMA) is still the most basic and most common material used in Prosthodontics. Polished and easy to use functionality, aesthetics, ease of maintenance and color stability, which led to the construction

of denture bases, a PMMA for an artificial prosthesis is used [1-3].

In addition to these advantages, it also has disadvantages such as low impact strength and low resistance to fatigue. Several studies indicate a very large number of resin bases breaks down after 2 to 3 years [4-7]. The main reason for this failure of the prosthesis falling on a hard surface and bending fatigue failure of the prosthesis under the affect it [8-12]. Various attempts have been made to avoid breaking the bass is like changing the chemical structure of the resin with the addition of cross linking agents or copolymerization [13-16]. The

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Received on: September 2016

Accepted after revision: October 2016

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innovative method for improving the physical properties of the prosthesis and increase its strength, a copolymer of styrene - methylmethacrylate was proposed. Styrene monomer is used to determine the composition of monomers in the presence of a radical polymerization of methylmethacrylate copolymer formed which in some ways is more favorable physical properties of the polymer poly methyl methacrylate [17-20].

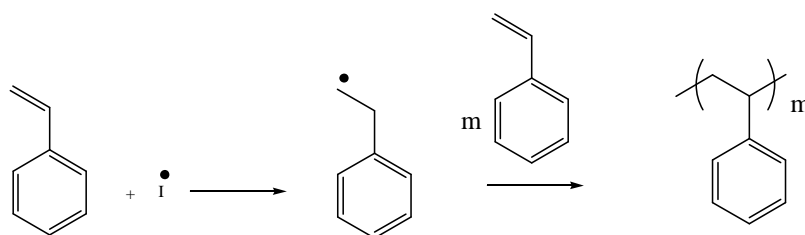
Experimental

General: Styrene (St) methylmethacrylate (MMA), methanol, benzoyl peroxide, and all the other chemicals and solvents were purchased and used as it was supplied from Merck (Germany). IR spectra of the compounds were obtained on a Shimadzu FTIR-8400s spectrometer using a KBr disk. The ^1H nuclear magnetic resonance (^1H NMR) spectra were

recorded on a Bruker AC 400 Advanced instrument at 400 MHz in dimethyl sulfoxide (DMSO-d₆) using tetramethylsilane as an internal standard. The glass transition temperature (T_g) was measured using thermal glass analysis (TGA),

Synthesis of polystyrene (PS) by free radical polymerization method

Styrene monomer (26 mmol) is removed and slowly added to the reaction flask. Benzoyl peroxide 0.107 g as a radical initiator reaction, we added to the flask, the reaction temperature is controlled at 60 ° C for 2 h was performed. PS cool down to room temperature for 12 hours. Obtain product was a clear crystal in weighted 1.3449 g (Yield: 71.3%). Synthesis of PS was shown in Scheme 1. PS characterized by FTIR, NMR, T_g and tensile strength physical test.

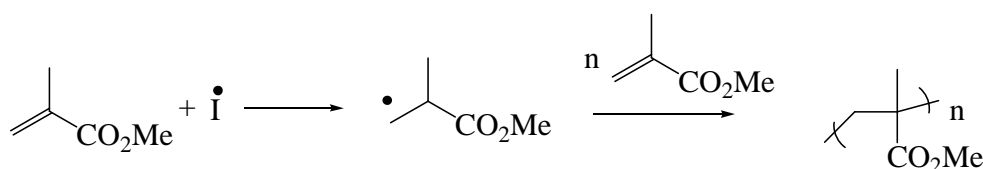


Scheme 1. Polystyrene polymerization process by free radical polymerization, Benzoyl peroxide radical initiator symbolized by I

Synthesis of poly methyl methacrylate (PMMA) by radical polymerization method

Methyl methacrylate (MMA) monomer 28.17 (mmol) is removed slowly added to the reaction flask. Benzoyl peroxide 0.107 g as a radical initiator reaction, we added to the flask, the reaction temperature is controlled at 60 ° C for

60 minutes was performed. PMMA cool down to room temperature for 24 hours. Obtain product was a clear crystal weighted 2.147 g (Yield: 72.4%). Synthesis of PMMA was shown in Scheme 2. The PMMA characterized by FTIR, NMR, T_g and tensile strength physical test.



Scheme 2. Poly methyl methacrylate polymerization by free radical polymerization, Benzoyl peroxide radical initiator symbolized by I

Synthesis of styrene / methyl methacrylate copolymer (St-Co-MMA) by radical copolymerization method

Radical copolymerization process is used Styrene and methyl methacrylate monomer with volume percent different amounts, the five synthesis is given below.

Synthesis of (St-Co-MMA) (20-80) using radical copolymerization

MMA (8 mmol) (80 wv. %) was added to St (9 mmol) (20 wv.%) in the reaction flask slowly. Benzoyl peroxide 0.134 g was added to a warm bath with a controlled temperature of 60 ° C for one hour was gently stirred. The copolymer to cool down to room temperature for 24 hours in a desiccator. Obtained copolymer was a clear crystal weighted 3.6885 g (Yield: 79.0%).

Synthesis of (St-Co-MMA) (40-60) using radical copolymerization

MMA (28 mmol) (60 wv. %) was added to St (17mmol) (40 wv. %) in the reaction flask slowly. Benzoyl peroxide 0.161 g benzoyl peroxide was added to the hot water bath controlled at 70 ° C was stirred gently for 45 minutes. The copolymer to cool down to room temperature for 24hr. Obtained copolymer was a clear crystal weighted 3.5249 g (Yield: 76.0%).

Synthesis of (St-Co-MMA) (50-50) using radical copolymerization

MMA (19 mmol) (50 wv. %) was added to St (17 mmol) (50 wv. %) in the reaction flask

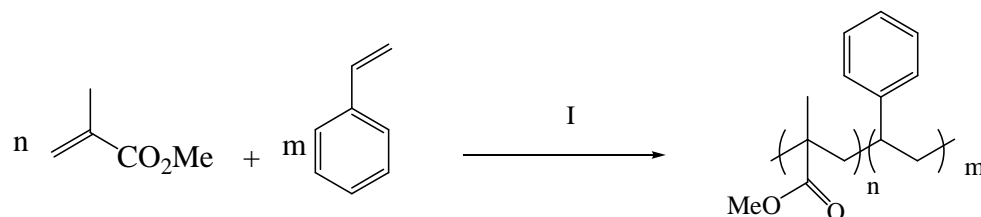
slowly. Benzoyl peroxide 0.215 g benzoyl peroxide was added to the hot water bath controlled at 60 ° C was stirred gently for 1h. The copolymer to cool down to room temperature for 24. Obtained copolymer was a clear crystal in weighted 2.2928 g (Yield: 62.0%).

Synthesis of (St-Co-MMA) (60-40) using radical copolymerization

MMA (19 mmol) (40 wv. %) was added to St (26 mmol) (60 wv. %) in the reaction flask slowly. Benzoyl peroxide 0.161 g benzoyl peroxide was added to the hot water bath controlled at 60 ° C was stirred gently for 1h. The copolymer to cool down to room temperature for 24hr. Obtained copolymer was a clear crystal weighted 3.5704 g (Yield: 77.5%).

Synthesis of (St-Co-MMA) (80-20) using radical copolymerization

MMA (9 mmol) (20 wv. %) was added to St (35 mmol) (80 wv. %) in the reaction flask slowly. Benzoyl peroxide 0.134 g benzoyl peroxide was added to a warm bath with temperature control at 60 ° C for 70 min was gently stirred. The copolymer to cool down to room temperature for 24. Obtained copolymer was a clear crystal weighted 2.9010 g (Yield: 63.4%). Styrene / methyl methacrylate copolymerization was shown in Scheme 3.



Scheme 3. styrene / methyl methacrylate copolymerization

Selected spectral data and physical tests

Polystyrene (PS): Clear crystal, FT-IR (KBr): 2847 cm^{-1} (stretching CH_2), 1600 cm^{-1} (stretching $\text{C}=\text{C}$); $T_g = 368 \text{ }^\circ\text{K}$; $F=70.3 \text{ N}$, $S=55.10 \text{ Mpa}$.

Selected spectral data and physical tests

Polymethylmetacrylate (PMMA): Clear crystal, FT-IR (KBr): 1245 cm^{-1} (stretching C-

O), 1725 cm^{-1} (stretching $\text{C}=\text{O}$); $T_g = 388 \text{ }^\circ\text{K}$; $F=82.64 \text{ N}$, $S=64.80 \text{ Mpa}$.

Selected spectral data and physical tests (St-Co-MMA):

Clear crystal, FT-IR (KBr): 2856 cm^{-1} (stretching CH_2), 720 cm^{-1} (bendind,op CH), 1728 cm^{-1} (stretching $\text{C}=\text{O}$); $^1\text{H-NMR}$ (FT-400 MHz, DMSO): δ : 3.60(s, a, 3H), 0.88 (s, b,3H), 1.82 (d, c, 2H), 1.10(d, e, 2H), 2.94(m, d,1H), 7.10 (d, f, 2H), 7.12 (t, g, 2H), 7.01 (t, h,

H); ¹³C NMR(400 MHz, DMSO): δ; 65.5 (a), 177.5 (b),45.3 (c), 19.3 (d), 20.2 (e), 49.3 (f), 23.1 (g), 145.7 (h),129 (i), 129.1 (j), 127.1 (k),. Scheme of St-Co-MMA for ¹H-NMR and ¹³C NMR were shown in figure4(a, b) respectively.

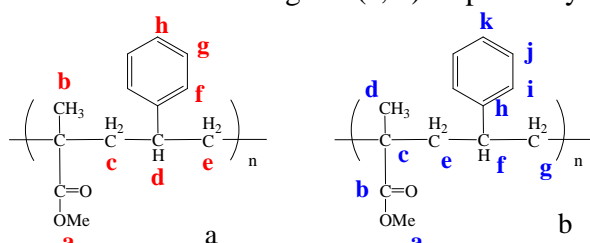


Figure 4. a, b, Scheme of St-Co-MMA for ¹H-NMR and ¹³C NMR

Results and discussion: Values T_g for St-Co-MMA samples 2 - 6 in table (1) is given.

Table 1. St-Co-MMA samples 2 – 6 , T_g Values

Samples	St-Co-MMA		T _g °K
	%St	%MMA	
2	20	80	384
3	40	60	380
4	50	50	378
5	60	40	376
6	80	20	372

Increase Styrene in St-Co-MMA from 20% to 40%, 50%, 60% and 80%, T_g changed to 384, 380, 378, 376 and 372 °K respectively. In other hand, it can be concluded that the increase wv% styrene in the copolymer is to reduce the T_g.

The innovative methods to improve the physical properties of the implant and strength of the St-Co-MMA was proposed. Using a combination of styrene monomer in free radical polymerization of monomers methyl methacrylate copolymer formed which in some ways is more favorable physical properties of the copolymer .

According to the standard tests for compressive strength specimens were tested synthetic copolymers, Figure (4) is a synthetic one on the tensile strength test based on the standard 1567 DIS / ISO studied. Synthetic samples before testing tensile strength, for two days in water 37 °C. Three-point was tested with a speed of 5 mm

per minute for each of the samples 1-7. The maximum force fractures samples were recorded. The tensile strength of the samples by Equation (1) was calculated.

$$S = \frac{l \times f \times 3}{h^2 \times b \times 2} \quad \text{Eq. 1}$$

In this equation, S tensile strength (MPa), f max force (N), l Distance between Reliance, b is the width of the sample and the sample thickness h. In equation (1) are the values of l, h, b, 65, 3.5 and 10 mm.

Table (4-2), maximum force fracture, tensile strength St-Co-MMA copolymers 1-7 were shown in Table (2).

Table 2. Tensile strength samples 1-7 St-Co-MMA

Sample	St %	MMA%	F (N)	S (MPa)
1	0	100	82.64	64.80
2	20	80	90.72	71.10
3	40	60	85.76	67.21
4	50	50	79.24	62.10
5	60	40	75.70	58.50
6	80	20	72.35	56.70
7	100	0	70.31	55.10

Sample (1) Only methyl methacrylate polymer formed from the start value of 64.80 MPa tensile strength (MPa) have been reported. The copolymer containing 20 wv. % styrene and 80% by weight of methyl methacrylate was tensail strength of the 71.10 (MPa) with increasing amounts of styrene was 40 wv. %, the amount of 3.9 MPa flexural strength decreased as the number of samples 67.21 (MPa) hit. Further increase to 50wv% styrene copolymer, tensile strength again fell to the 62.10 (MPa) were reported. With the increased weight of the copolymer of styrene at 60 and 80 wv. %, tensile strength 58.50 and 56.70 (MPa) was obtained respectively. In the sample (7) that only Homopolymer styrene is also used to measure the tensile strength and the lowest 55.10 (MPa) with the organization. The sample (7) no traces of PMMA. We can say that the best of the above samples, samples, (2) that the tensile strength 71.10 (MPa) has shown the highest strength and

it combines a copolymer of styrene methyl methacrylate (20-80) wv. %.

Conclusion: We can say that samples, (2) are the best of the above samples, that the tensile strength 71.10 (MPa) has shown the highest tensile and it combines a copolymer of styrene methyl methacrylate (20-80).

Acknowledgments: The authors are grateful to Payame Noor University for receiving financial support.

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