

INFLUENCE OF CONCENTRATIONS OF METHACRYLATE AND ACRYLATE MONOMERS ON THE PROPERTIES OF FIBER REINFORCED POLYMETHYL METHACRYLATE DENTURE BASE MATERIALS

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Abstract: The study aimed to evaluate the effects of adding different concentrations of 2 hydroxyethyl methacrylate (HEMA), 2-hydroxyethyl acrylate (HEA), ethyl methacrylate (EA) and isobutyl methacrylate (IBMA) monomers on the structural, thermal and mechanical properties of a fiber reinforced heat-polymerized acrylic resin. For each test, 126 acrylic resin specimens were fabricated and divided into 6 groups with 7 specimens each. One group was the control group, the other one is a fiber reinforced group and others were the test groups, which were formed according to the different concentrations of monomers. 6 mm length, and the weight ratio of 3% short glass fibers are added to acrylic powder polymerized by heating. The 2%, 5%, 10%, at 20 % ratios of different comonomers added to a monomer of MMA are composed of copolymer structures. Flexural strength was assessed with a three-point bending test using a universal testing machine. Impact strength testing was conducted using an impact test machine by the Charpy method. The analysis of the connection between acrylic resin and fiber by SEM and structural changes in the acrylic resin was investigated by FTIR spectroscopy. Data analyses using analysis of Kruskal-Wallis and Mann-Whitney U tests ($\alpha=0.05$) significant difference tests showed that adding 2%, 5% HEMA and IBMA monomers significantly increased the

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flexural and impact strength compared to the control, only fiber and others group ($P < 0.05$). It is observed that the process of adding low concentration of HEMA and IBMA monomers improved certain mechanical properties of fiber reinforced with polymethylmethacrylate.

Keywords: Denture, base materials, acrylic resin, methacrylate, acrylate, fiber reinforced

Introduction

The most commonly used acrylic resin in preparation of denture base material is the polymethylmethacrylate (PMMA). The acrylic resins have been used extensively for the fabrication of denture bases, because of their aesthetic qualities and reparability, but some properties of denture base resins, such as dimensional stability, porosity, and mechanical strength may play a significant role in the clinical performance of complete dentures and removable partial prostheses.¹⁻³

Denture fractures in clinical use may result from sizeable transitory force, caused by an accident or a low force during repeated chewing.⁴

Researchers have attempted to improve the mechanical properties of polymethyl methacrylate, and this material has been subjected to numerous different procedures, including several types of reinforcement materials. The literature reports reinforcement with various fibers including carbon, aramid and glass fibers.^{5,6}

Different polymers have been developed to be used as denture base resins, to overcome some of the mechanical deficiencies of poly(methyl methacrylate) (PMMA) such as polyamides, epoxy resin, polystyrene, vinyl acrylic, rubber graft copolymers, polycarbonates and nylon.

Stafford and Smith have investigated polycarbonates and found that these materials have encouraging possibilities as a denture base because they were found to be superior to the acrylic resin in deflection, impact strength, and thermal expansion.⁶

To strengthen the acrylic structure, they have created new matrix constructions for strengthening chemical structures by adding different copolymers with/without fiber addition. Several types of monomers have been added to MMA and PMMA. Few studies show that PMMA became stronger with some methacrylate monomers.^{8,9} Ethyl methacrylate, butyl methacrylate, butyl acrylate, tetramethyldisiloxane, norbornyl and phenyl methacrylate monomers have been used with MMA to produce a modified copolymer structure, but conflicting results have been reported.^{10,11} Both 2-hydroxyethyl methacrylate (HEMA) and isobutyl methacrylate (IBMA)^{8,9} were used to improve the mechanical properties of PMMA. Johnson and Jones studied the mechanical properties of PMMA and copolymers with ethyl and butyl methacrylates. Mechanical properties of PMMA decreased when increasing the concentration of butyl and ethyl methacrylates.⁸ Moreover, heat-polymerized acrylic resin powder was mixed separately with tert-butyl, ethyl, and isobutyl methacrylate monomers.¹² They found that there was no difference in tensile strengths between the polyacrylates and the control group.

The aim of this study was to evaluate some properties such as mechanical (transverse strength and impact strength) spectroscopic and thermal properties and surface morphology of glass fiber reinforced acrylic resin with the ratios of different monomers incorporating to PMMA. We hypothesized that the addition of some hydrophobic or hydrophilic monomers to PMMA based denture base resins would improve the mechanical properties of the resin.

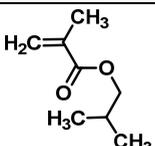
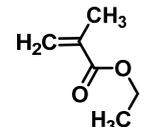
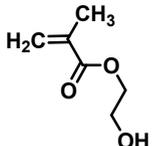
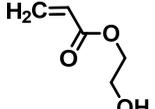
Results and Discussion

The acrylic resin is the most commonly used material for the construction of denture base material, but this material is not perfect

because of its relatively low mechanical strength, which can cause the fracture of the denture.

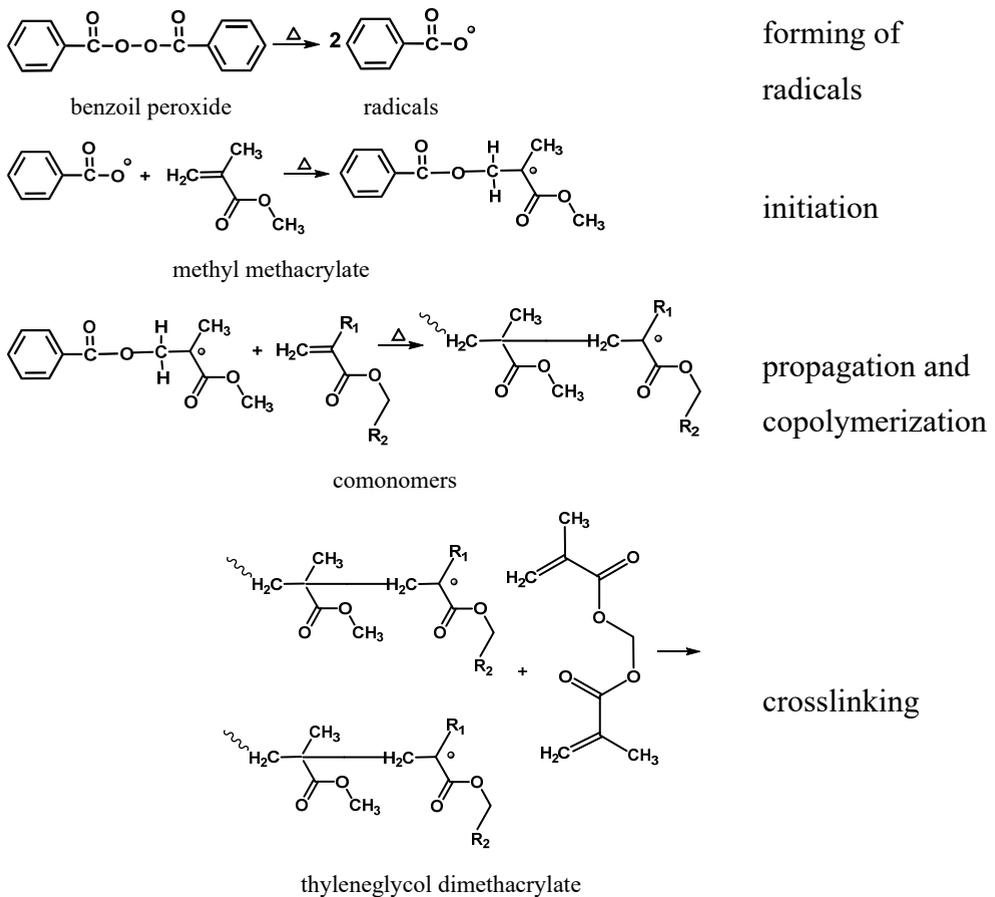
In this study investigated the effects of some comonomers, such as isobutyl methacrylate [IBMA], ethyl methacrylate [EMA], 2 hydroxyethyl methacrylate [HEMA] and 2 hydroxyethyl acrylate [HMA], (Table 1), on impact strength and flexural strength of acrylic resin. At the same time, the glass fiber was used because the impact strength, elasticity modulus, and transverse resistance of the acrylic resin increased. The glass fiber was also preferred from aesthetic reasons (nice white color) and because it has more homogeneous distribution than other fibers in the reaction phase.¹³⁻²⁰ Since the use of low-concentration and short-length fibers strengthens the mechanical properties of acrylic resins, 6 mm long and 3 wt% glass fiber is used.

Table 1. Comonomers used in this study.

Comonomer	Abbreviation	Chemical structure	Molar mass / g mol ⁻¹
Isobutyl methacrylate	IBMA		142.20
Ethyl methacrylate	EMA		114.14
2 Hydroxyethyl methacrylate	HEMA		130.14
2 Hydroxyethyl acrylate	HEA		116.12

Synthesis of Acrylic resin containing copolymer

Acrylic resins composed of PMMA, MMA, and comonomer such as EMA or IBM or HEA or HEMA with or without the glass fibers were prepared by heating, via free radical solution polymerization, using EGDMA as a crosslinking agent. The polymerization mechanism is very well established and schematic representation is given in Figure 1. It is evident that the polymerization and crosslinking process took place simultaneously.



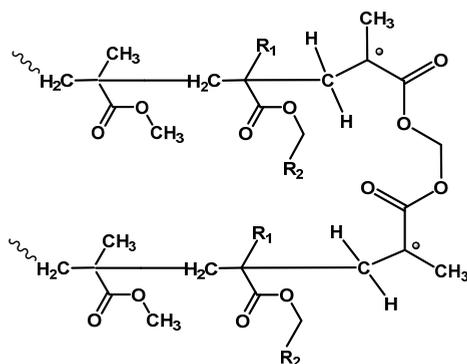


Figure 1. Possible crosslinking mechanism of copolymers, HEA; R₁: - H, R₂: - CH₂OH, HEMA; R₁: - CH₃, R₂: - CH₂OH, EM; R₁: - CH₃, R₂: - CH₂CH₃, IBM; R₁: - CH₃, R₂: - CH₂ C (CH₃)₂.

The processed molds were left to cool at room temperature for 20 min, then placed under running tap water for 10 min. Subsequently, the specimens were excluded from the mold and excess material trimmed. All samples were stored in distilled water at 37°C for two weeks before tests.

Spectroscopic analysis of the acrylic resin

The typical FTIR spectra of acrylic resin with the comonomers without glass fibers are shown in Figure 2.

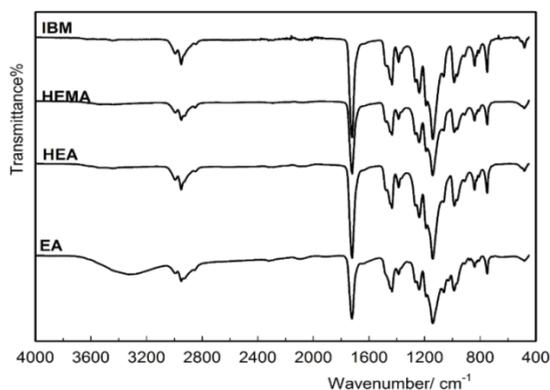


Figure 2. FTIR spectra of acrylic resin containing comonomers without fiber.

The spectra in Figure 2 are similar, because the chemical structures of the polyacrylic copolymers given in Table 5 and those used in the present study are also similar. According to FTIR analyses,²¹ the bands in 1100–1400 cm^{-1} region are assigned to the bending mode of the C–C–O, and C–C=O bonds belonging to esters in the backbone chain. The carbonyl (C=O) bands in the polymers are 1725–1720 cm^{-1} . The significant signals at 2920–2850 cm^{-1} were due to C–H vibrations.

Thermal analysis

TG analysis

The thermal degradation parameters such as the initial degradation temperatures (T_i), the temperature of maximum speed (T_{max}), the degradation final temperature (T_f), the half-life temperature (T_h), the maximum decomposition rate (r_{max}) and the maximum mass loss at the maximum decomposition rate (C_{max}) values, calculated from the thermograms of acrylic resin with comonomers, without the glass fibers (Figure 3 and 4), are given Table 2.

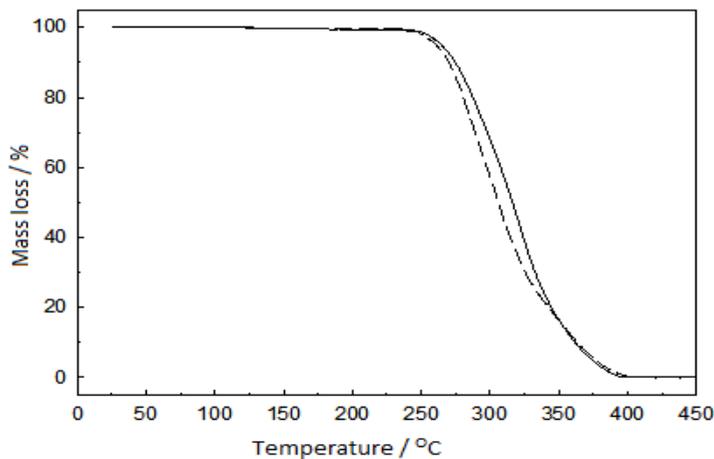


Figure 3. Thermal degradation of acrylic resin containing hydrophobic comonomers without fiber: —; IBMA, - - ; EMA.

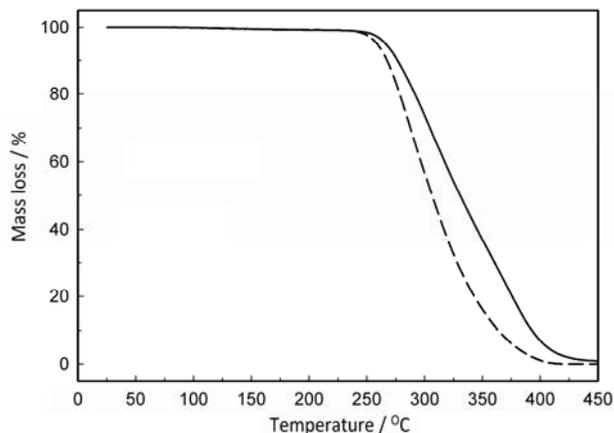


Figure 4. Thermal degradation of acrylic resin containing hydrophilic comonomers without fiber: —; HEA, - - ; HEMA.

Table 2. Thermal degradation parameters and Glass transition temperature of the copolymers.

Copolymer	T_i °C	T_{max} °C	T_f °C	T_h °C	r_{max} mg min ⁻¹	C_{max} %	T_g °C
MMA/IBMA	262.05	301.82	391.87	306.16	1.12	41.88	219.45
MMA/EMA	269.38	321.65	380.77	315.96	0.92	42.79	229.52
MMA/HEMA	259.98	288.06	391.32	306.33	0.93	42.96	231.92
MMA/HEA	264.23	311.94	406.58	330.58	0.97	43.21	234.80

The thermograms of acrylic resins exhibit only one distinct degradation stage in the range of 260/265 - 390/405 °C. The thermal degradation of acrylic resins in this temperature range is the chain radical reaction of depolymerization²²⁻²³. The initiation of acrylic resins depolymerization proceeds via a statistic rupture of polymer, with the formation of two free macroradicals, via disruption of the bonding near the terminal double bond, with the formation of an active macroradical and a relatively stable allyl radical.

DSC Analysis

The glass transition temperatures²⁴ of the acrylic resins containing the comonomers without the glass fibers were determined from the DSC thermograms (Figure 5 and 6 and Table 2).

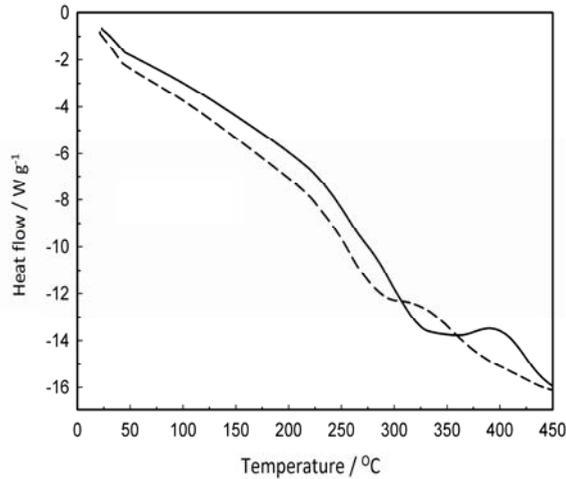


Figure 5. DSC Thermograms of acrylic resin containing hydrophobic comonomers without fiber: —; IBMA, - - - ; EMA.

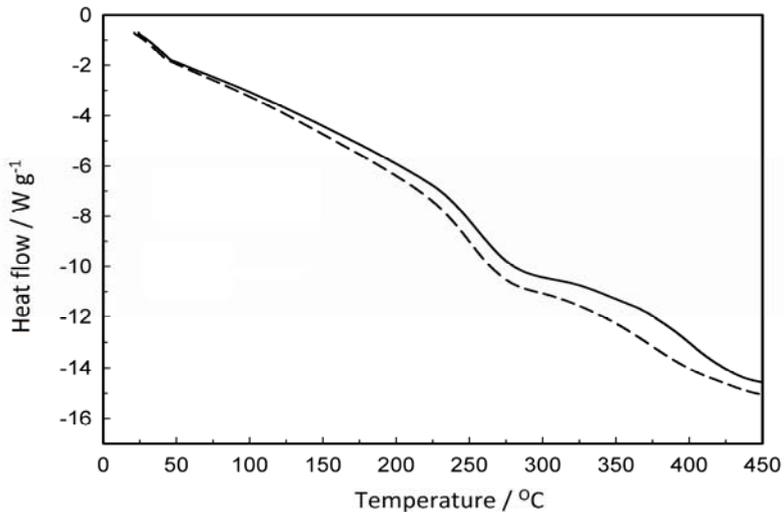


Figure 6. DSC Thermograms of acrylic resin containing hydrophilic comonomers without fiber: —; HEA, - - - ; HEMA.

The glass transition temperatures (last column in Table 2) of the acrylic resins containing hydroxyl group were higher than the glass transition

temperature for the other resins. So, the increase in the glass transition is related to the hydroxyl group in acrylic resin, due to the hardening effect of the hydrogen bonding ability of hydroxyl group from the acrylic resin.

Water sorption

The values of water sorption capacities (WS)²⁵ of the test samples of polymers containing acrylate comonomers in the concentration of 2, 5, 10 and 20 % were calculated, and the graphs are presented in Figure 7.

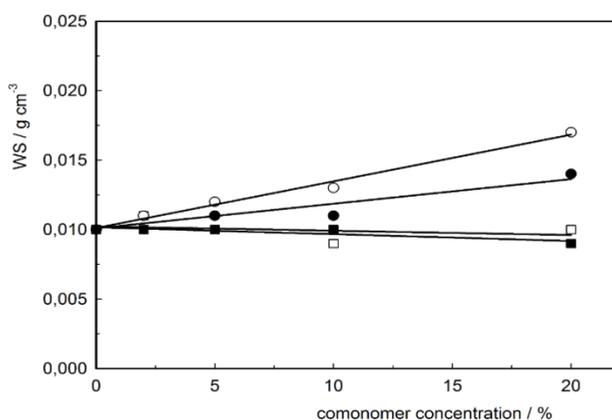


Figure 7. The change of water sorption of acrylic resin with the comonomers concentration: ○; HEA, ●; HEMA, ■; IBMA, □; EMA.

While the WS values of the polymer containing hydrophilic comonomers (i.e., HEA and HEMA) slowly increased with the concentration, the ones of the others remained approximately constant.

Morphological analysis

The surfaces of acrylic copolymers with fiber samples were examined by scanning electron microscopy in two different magnifications (Figure 8-13).

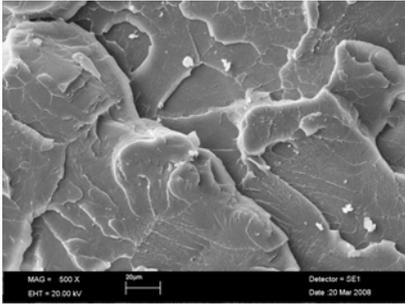


Figure 8. Fractured surface of a control group under SEM (X 500 magnification).

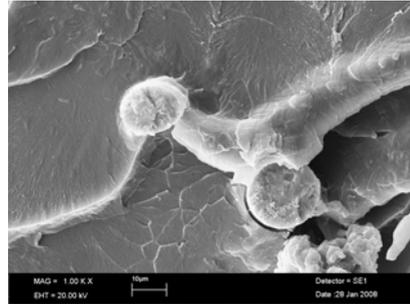


Figure 9. Fractured surface of only fiber group under SEM (X 1000 magnification).



Figure 10. Fractured surface of fiber + HEMA group under SEM (X 1000 magnification).

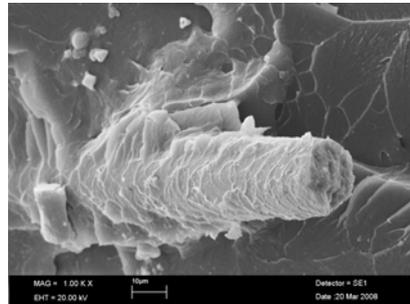


Figure 11. Fractured surface of fiber + IBMA group under SEM (X 1000 magnification).

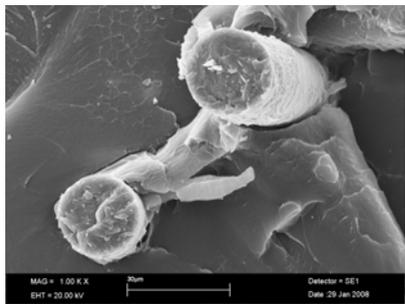


Figure 12. Fractured surface of fiber + HEA group under SEM (X 1000 magnification).

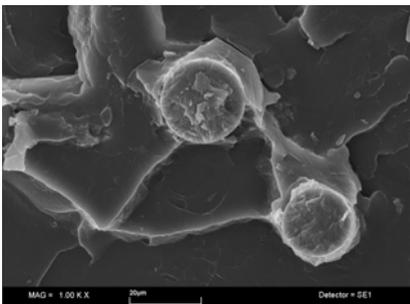


Figure 13. Fractured surface of fiber + EA group under SEM (X 1000 magnification).

In the SEM examination of the copolymers groups, the good compatibility between fibers and acrylic copolymers with HEMA and IBMA was observed, while spaces between fibers and copolymers with HEA and EMA and acrylic residues appear at the surfaces of the fibers.

Therefore, it can be said that acrylic copolymers with HEMA and IBMA are more compatible for the acrylic copolymers than HEA and EMA.

Mechanical test

The means and standard deviations of the flexural and impact strength²²⁻³⁰ of the control, fiber only and different concentration of copolymer groups are given in Tables 3 and 4.

Table 3. Flexural strengths (MPa) in each group of specimens (n = 7) presented as means (X) and standard deviations (Sd).

Groups	% 2 X±Sd	% 5 X±Sd	% 10 X±Sd	% 20 X±Sd	Friedman Wilcoxon
Control	88.88±4.69	88.88±4.6	88.88±4.69	88.88±4.69 ^a	
Fiber Group	95.21±9.75	95.21±9.75	95.21±9.75	95.21±9.75	
Fiber + IBMA	130.79±14.1 6	106.97±15.1 0	104.34±9.72	100.43±6.5 1	p = 0.011 * p < 0.05
Fiber + HEMA	110.62±2.91	114.29±4.81	104.86 ±11.06	104.06±10. 76	p = 0.011 * p < 0.05
Fiber + HEA	83.94±8.21	87.71±3.34	83.78±8.24 ^f	73.37±5.45	p = 0.034 * p < 0.05
Fiber + EMA	94.49±7.78	100.43±7.02 g	95.69±7.77	100.49±6.2 4	p = 0.366 p > 0.05
	KW = 32.77 p = 0.000 * p < 0.05	KW = 24.99 p = 0.000 * p < 0.05	KW = 26.43 p = 0.000 * p < 0.05	KW = 25.18 p = 0.000 * p < 0.05	

Table 4. Impact strengths (J m⁻²) in each group of specimens (n = 7) presented as means (X) and standard deviations (Sd).

Groups	% 2 X±Sd	% 5 X±Sd	% 10 X±Ss	% 20 X±Ss	Friedm an Wilcoxo n
Control	9250.00±851.1	9250.00±851.1	9250.00±851.1	9250.00±851.1	
Fiber Group	10452.38±1353	10452.38±135	10452.38	10452.38±135	
Fiber + IBMA	12497.22±1759	10880.95±254 7.40	8654.76±2497. 53	5708.33±2254. 11	p = 0.009 * p < 0.05
Fiber + HEMA	11005.86±1104	10291.67±170 5.96	11416.67 ±2311.78	7619.04±2423. 77	p = 0.016 * p < 0.05

Table 4. Continued

Fiber	+ 10464.29±2065	10392.86±159	9642.85±2059.	9297.61±2078.	p = 0.045
HEA	31	5.14	48	80	p < 0.05
Fiber	+ 10142.86±2089	10483.33±173	9839.28±2718.	10547.67±118	p = 0.116
EMA	27	1.7	86	2.53	p > 0.05
	KW = 14.25	KW = 15.25	KW = 4.70	KW = 16.83	
	p = 0.014	p = 0.009	p = 0.453	p = 0.005	
	p < 0.05	p < 0.05	p > 0.05	p < 0.05	

Significant interactions were observed between the control, fiber only and copolymer groups with respect to flexural and impact strength according to the Kruskal Wallis³¹ ($p < 0,05$).

The highest tensile strength was observed for the 2% IBMA group (130,79±14,16 MPa) and showed a significant increase in transverse strength compared to the control and fiber only group ($p < 0.05$), whereas the 20% HEA group appeared to have the lowest value (73,37±5,45 MPa).

Increased mean impact strengths were observed after reinforcing all acrylic resin specimens with only fiber and 4 monomers. Only samples reinforced with 2 % IBMA (12497,22±1759,45 MPa) showed however a significant increase in impact strength compared to the control and fiber only group ($P < 0.05$), whereas the 20% IBMA group appeared to have the lowest value (5708,33±2254,11 MPa) not to a significant level ($P > 0.05$).

In the light of these findings, the values of flexural and impact strength of the samples from control group, control group with fibers and test groups indicate that the measured values of the acrylic polymers containing HEMA and IBM in low concentrations were increased.

Cho and et al.,³² adding butyl methacrylate to strengthen poly (methyl methacrylate), have found that the poly (butyl methacrylate) group with functional end groups was more resistant in the transverse strength test. As a result of these findings, they reported that van der Waals interactions between molecules were effective and that these interactions would make

the material more resistant. Clarke³³ reported that, when alkyl groups (such as $-\text{CH}_2\text{CH}_3$, $-\text{CH}_2\text{CH}_2\text{CH}_3$, $-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$) containing a higher number of carbons are incorporated into the PMMA ester, there will be a decrease in the relaxation temperature and impart flexibility of the molecule.

Based on these studies,^{32,33} we can also say that the van der Waals interaction is stronger in our work, based on the resistivity increase of the acrylic resins in the case of including IBMA and HEMA monomers in the preparation, due to the interaction between their alkyl groups and the acrylic resin. Moreover, IBMA and HEMA containing more carbon atoms than other monomers, conferred flexibility to the copolymer structure and increased the resistance of the resin.

Experimental

Materials

The monomers and all reagents of chemically pure or analytical grade were used as received. E-Glass fiber (WR3-2400) was gifted from Cam Elyaf Sanayi (Kocaeli/Türkiye). Heat-cured acrylic resin (PMMA) (containing % 0.05 benzoyl peroxide) and methyl methacrylate (MMA) (containing 10% ethylene glycol dimethacrylate) were obtained from Meliodent Bayer Dental (Germany). Monomers such as ethyl methacrylate (EMA) and isobutyl methacrylate (IBMA) and 2-hydroxy ethyl acrylate (HEA) were obtained from Fluka, Sigma Aldrich Chemie GmbH, and 2-hydroxy ethyl methacrylate (HEMA) was purchased Merck, Schuchardt OHG. Some properties of the comonomers are listed in Table 1.

Preparation of the acrylic resin

For the preparation of fiber reinforced PMMA based denture base resins with some comonomers, the glass fibers 3% (mass) and 6 mm in length were soaked in monomer in a Petri dish for better bonding of these fibers with the PMMA resin matrix except for the control group. The fiber amounts to be added were determined relative to the premixed, measured resin powder mass.

For the test groups, each of the hydrophobic comonomers such as IBMA, EMA and hydrophilic comonomers such as HEMA, HEA groups were mixed separately to the acrylic resin liquid at 2, 5, 10, 20 % by mass, respectively, prior to polymerization.

The compositions of control and test groups were shown in Table 5.

Table 5. Groups and different comonomers used in this study.

Group	
1	Control group (without fiber and comonomer)
2	Only fiber group (3% glass fiber added)
3	3% fiber and 2, 5, 10, 20 % HEMA (2 hydroxyethyl methacrylate) added group
4	3% fiber and 2, 5, 10, 20 % EMA (Ethyl methacrylate) added group
5	3% fiber and 2, 5, 10, 20 % IBMA (Isobutyl methacrylate) added group
6	3% fiber and 2, 5, 10, 20 % HEA (2 hydroxyethyl acrylate) added group

126 wax specimens were prepared for the tests, 7 for each group mentioned in Table 5, with the following dimensions: impact strength (50x6x4 mm) according to ISO 1567:1988 and flexural strength (65x10x2.5

mm) test according to ADA Standard No 12. Then, all polymers were flaked and eliminated in a conventional manner.

The powder/liquid ratio of 2.34 g mL^{-1} was maintained as recommended by the manufacturers for the control groups. The glass fiber was added to acrylic resin powder at 3% concentration by weight for the only fiber groups. In the copolymer groups, preparation was carried out at different monomers with 2%, 5%, 10% and 20% according to the molar mass ratio, respectively. First, each of the comonomers was added to liquid MMA monomer, and dissolved monomer mixture was obtained. Then, the fiber with powder resin was added to the dissolved liquid and experimenters waited for the mixture to become pasty. The acrylic pasty was infused into space. The flask was closed and placed under hydraulic pressure. The specimens were then polymerized according to manufacturer's instructions (2 h in boiling water). The processed molds were left to cool at room temperature for 20 min and then placed under running tap water for 10 min. After the specimens were excluded from the mold and trimmed excess material.

All specimens were stored in distilled water at $37 \text{ }^{\circ}\text{C}$ for two weeks before tests.

Spectroscopic analysis of acrylic resin

FTIR spectra of all polymers containing 20% comonomer were recorded with FTIR Nicolet-520 spectrophotometer in the $4000 - 400 \text{ cm}^{-1}$ range, on grinded polymer pellet with KBr, and 30 scans were taken at 4 cm^{-1} resolution.

Thermal analysis

Thermal analysis of the polymers was carried out using TG and DSC (Shimadzu-50 model Thermal Analyzer). Thermogravimetric and differential scanning analyses were performed employing 10 mg samples in

a platinum pan heating up to 450 °C under a nitrogen gas flow rate of 25 mL min⁻¹ with a heating rate of 10 °C min⁻¹.

Water sorption

Specimens were kept in a desiccator at 37±1 °C for 24 hours. This mass value was considered the initial mass of the specimen (m_0). All samples were added in a distilled water-filled beaker and weighed daily on an analytical scale with an accuracy of 0.0001 g, in a water bath maintained at 37°C, until a constant mass (m_e) was achieved.

The values for water sorption (WS) in g cm⁻³ for each of the specimens were calculated using the following equations:

$$\text{WS \%} = \frac{m_e - m_0}{V}$$

Where m_0 is the conditioned mass, in g, prior to immersion in water; m_e is the mass of the specimen in g, after immersion in water, and V is the volume of the specimen in cm³.

Morphological analysis

The fractured surfaces of glass fiber reinforced with heat-cured acrylic resin samples were coated with a layer of 5 nm-thick gold, and then SEM photographs were recorded with LEO 440 Scanning Electron Microscopy (Zeiss, Oberkochen, Germany) at the magnifications of 500x and 1000x.

Mechanical test

Mechanical tests of glass fiber reinforced with a heat-cured acrylic resin such as impact strength and flexural strengths were carried out in this study. For the tests, 126 acrylic resin specimens were fabricated and divided into 6 groups (18 sample types, Table 5) with 7 specimens each. One group was the control group; the other one is the fiber reinforced group and the third was the test group, formed according to the different concentrations of

fibers incorporated into the acrylic resin (Table 2). Finally, different comonomer materials (seven specimens for each group) were prepared for impact and transverse strength tests, respectively.

Each group was subjected to flexural strength test using a universal testing machine (Lloyd LF Plus; Ametek Inc, Lloyd Instruments, Leicester, UK), at a crosshead speed of 5 mm min⁻¹. The impact strength test was performed according to ISO Standard 1567:1988,¹¹ using an impact test machine (Coesfeld, Impact tester, Germany) by the Charpy method with a pendulum of 0.5 J, in which the specimens were horizontally positioned with a distance of 40 mm between the two fixed supports.

The flexural strength F_s was calculated from the formula;

$$F_s = \frac{3Fl}{2bh^2}$$

where F is the maximum load (N), l the span length (mm), b the width of the test specimen (mm) and h the thickness of the test specimen (mm).

Impact strength (I_s) of the specimen was calculated by using the following formula;

$$I_s = \frac{AE}{bh}$$

where AE is the absorbed energy (J), b the width of the test specimen (mm) and h the thickness of the test specimen (mm).

Statistical Analysis

The flexural and impact strengths (means and Standard deviations) between different concentration for the same monomer group were statistically evaluated by Friedman and Wilcoxon tests ($\alpha=0.05$) and the ones between different monomer for the same concentration were statistically evaluated by Kruskal-Wallis and Mann-Whitney U tests ($\alpha=0.05$), by using a SPSS statistical software program (version 15.0, SPSS Inc., Chicago, IL, USA).

Conclusion

Under the conditions of the present study, the following conclusions may be drawn:

- Since denture fractures are frequently encountered in clinical use, it is aimed to improve the mechanical properties of acrylic resins obtained by adding glass fiber and various monomers to this study. In accordance with this purpose, some properties (ie., mechanical (transverse strength and impact strength), spectroscopic, thermal properties, water sorption, and surface morphology) of glass fiber reinforced acrylic resin with the ratios of different monomers incorporating to PMMA were evaluated.
- The FTIR spectra of the prepared materials showed that the incorporation of acrylic comonomers such as ethyl methacrylate, isobutyl methacrylate, 2-hydroxy ethyl acrylate and 2-hydroxy ethyl methacrylate to PMMA created the copolymer structure
- The highest flexural and impact strength was obtained by the addition of 2% IBMA monomer.
- PMMA prepared by adding a low concentration of HEMA and IBMA increased the impact and flexural strength values of PMMA denture base resins among the trial groups.
- A suitable adhesion between fiber/resin surface was indicated by SEM images, except for HEA and EM monomers.

As a result, we can conclude that transverse and impact strength of fiber-reinforced acrylics increased by the addition of HEMA and IBM comonomers at low concentrations. So it may be suitable to use these monomers with fiber-reinforced acrylics.

Acknowledgments

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