RESEARCH

Evaluation of the mechanical properties and residual monomer content of copolymer acrylic resins polymerized by different methods

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ABSTRACT

Evaluation of the mechanical properties and residual monomer content of copolymer acrylic resins polymerized by different methods

Background: The aim of this study was to evaluate the effect of adding four different monomers on the flexural strength, elastic modulus, impact strength and residual monomer content of denture base resins polymerized by copolymerization mechanism.

Methods: Butyl methacrylate (BMA), Isobutyl mathacrylate (IBMA), 2-hydroxyethyl methacrylate (HEMA) and methacryl-polyhedral silses quioxane (POSS-MA) were added to monomers of conventional heat (HP) and microwave polymerized (MP) polymethyl methacrylate (PMMA) resin at the concentrations of 2%, 5% and 10% per volume. The flexural strength, elastic modulus and impact strength of specimens were measured with an universal testing machine and Charpy-type impact tester. Residual monomer contents were determined by high performance liquid chromatography (HPLC) method.

Results: 10% IBMA and 10% HEMA enhanced the flexural strength of HP resin. For the elastic modulus of MP resin, significant differences observed between the control group and 2 and 5% IBMA, 5 and 10% HEMA, and 5% POSS-MA groups ($P<0.05$). Copolymerization mechanism had no positive effect on the impact strength of denture base resins. Significant differences were observed between the residual monomer of control group and 10% IBMA, 10% BMA, 10% POSS-MA groups of HP resin ($P<0.05$). In MP resin group significant differences were observed between control group and 5% IBMA, 2-5-10% HEMA groups ($P<0.05$).

Conclusion: Copolymerization process was effective on the flexural strength of HP denture base resin with 10% IBMA and 10 % HEMA. However the process caused an increase in the residual monomer content of some resin groups.

KEYWORDS

Copolymer, denture base resin, flexural strength, impact strength, residual monomer.

ÖZ

Farklı yöntemlerle polimerize edilen kopolimer akrilik rezinlerin mekanik özelliklerinin ve artık monomer içeriğinin değerlendirilmesi

Amaç: Bu çalışmanın amacı dört farklı monomer eklenerek kopolimerizasyon mekanizmasıyla polimerize olmuş protez kaide rezinlerinin bükülme dayanımı, elastik modulus, çarpma dayanımı ve artık monomer içeriğinin değerlendirilmesidir.

Gereç ve Yöntemler: Bütil metakrilat (BMA), isobütil metakrilat (IBMA), 2-hidroksietil metakrilat (HEMA) ve metakril-polihedral silseskioksan (POSS-MA) hacimce %2, %5 ve %10 konsantrasyonlarında konvansiyonel ısı (HP) ve mikrodalga (MP) akrilik monomerlerine ilave edilmiştir. Universal test cihazı ve Charpy tip çarpma test cihazı ile örneklerin bükülme dayanımı, elastik modulusu ve çarpma dayanımı ölçülmüştür. Yüksek performanslı sıvı kromatografisi (HPLC) metoduyla artık monomer içerikleri belirlenmiştir.

Bulgular: %10 IBMA ve %10 HEMA, HP rezinlerin bükülme dayanımını artırmıştır. MP rezin grubunun elastik modulusu için kontrol grubu ile %2 ve %5 IBMA, %5 ve %10 HEMA ve %5 POSS-MA grupları arasında anlamlı farklılıklar gözlenmiştir ($P<0.05$). Kopolimerizasyon mekanizmasının protez kaide rezinlerinin çarpma dayanımı üzerinde olumlu bir etkisi olmamıştır. HP rezin kontrol grubu ile % 10 IBMA, % 10 BMA, %10 POSS-MA grupları arasında artık monomer bakımından anlamlı farklılıklar saptanmıştır ($P<0.05$). MP rezinde ise kontrol grubu ile %5 IBMA ve %2-5-10 HEMA grupları arasında anlamlı farklılıklar gözlenmiştir ($P<0.05$).

Sonuç: Kopolimerizasyon işlemi %10 IBMA ve %10 HEMA eklenmiş HP protez kaide rezinlerinin bükülme dayanımında etkili olmuştur. Bununla birlikte işlem, bazı rezin gruplarının artık monomer miktarında artışa neden olmuştur.

ANAHTAR KELİMELER

Kopolimer, protez kaide rezini, bükülme dayanımı, çarpma dayanımı, artık monomer.

Polymethyl methacrylate (PMMA) is a widely used material in the construction of removable partial and complete dentures.[1] Since the conventional processing technique requires polymerization in a water bath, microwave polymerization technique, that was introduced by Nishii[2], allows to a faster and easier processing method in a microwave oven.[3]

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Flexural fatigue and impact forces cause to denture fracture. While the flexural fatigue failures occur by repeated flexural loads and consequently development of microscopic cracks in stress concentration areas, impact failures usually occur as a result of sudden blow to the denture and accidental droppings.[1] To improve the mechanical properties of denture base polymers, several attempts such as addition of metal wires[4] and different types of fibers[5], generating chemical modification by using copolymerization mechanism[6], have been reported.

Copolymerization is the process that provides a new polymeric material called copolymer by linking more than one monomer having different physical properties and volume ratios.[6] In recent studies various types of monomers such as acrylamide, butadiene styrene, fluoroalkyl methacrylate, ethyl methacrylate (EMA), butyl methacrylate (BMA), isobutyl methacrylate (IBMA), 2-hydroxy ethyl methacrylate (HEMA), and polyhedral silsesquioxane (POSS) have been added to methyl methacrylate (MMA) monomer to produce a modified copolymer structure of PMMA resins and contradictory results have been reported.[3,6-10] Depending on the type of denture base resin, curing method, polymerization type and, temperature the conversion of the monomer to the polymer is not complete and some residual monomer left in the polymer.[11] Residual monomer is an irritant for oral tissues, acts as a plasticizer and affects the mechanical and physical properties of denture base resins.[12] Gas chromatography, infrared spectroscopy, high performance liquid chromatography (HPLC) and fluorescent flow injection methods have been used to detect the residual monomer content of acrylic resin materials.[12-15]

The object of this *in vitro* study was to evaluate the effect of four different monomer supplements on the flexural strength, elastic modulus, impact strength and residual monomer content of denture base resins polymerized by copolymerization process.

MATERIALS AND METHODS

In the present study two PMMA-based acrylic resins; (1) conventional heat-polymerized (HP) resin (Meliodent, Bayer Dental, Newbury, Berkshire, United Kingdom), (2) microwave- poymerized (MP) resin (Acron MC, GC Dental, Tokyo, Japan) and four different monomers; (1) IBMA (Sigma Aldrich GmbH, Germany), (2) BMA (Sigma Aldrich GmbH, Germany), (3) HEMA (Merck Schuchardt, Munich, Germany), (4) Methacryl-POSS (POSS-MA) (Sigma Aldrich GmbH, Germany) were used.

A total of 390 wax patterns were prepared by using stainless steel molds with dimensions of 65x10x2.5 mm (according to ADA specification no.12)[16] for flexural strength test, 50x6x4 mm (according to ISO/DIS 1567 international standard)[17] for impact strength test and 20 mm diameter and 2 mm thickness for residual monomer content test.

IBMA, BMA, HEMA and POSS-MA monomers were added to the MMA monomer of both HP and MP resins with a milimeter syringe at the percentages of 2%, 5% and 10% per volume and were bottled separately. The monomer mixtures of specimen groups were expressed in Table 1.

Table 1.

Monomer mixtures of conventional heat and microwave polymerized resin specimen groups

Monomer mixtures
100% MMA (Control)
2% IBMA-98% MMA
5% IBMA-95% MMA
10% IBMA-90% MMA
2%BMA-98% MMA
5% BMA-95% MMA
10% BMA-90% MMA
2% HEMA-98% MMA
5% HEMA-95% MMA
10% HEMA-90% MMA
2% POSS-MA-98% MMA
5% POSS-MA-95% MMA
10% POSS-MA-90% MMA

All of the specimens were wet ground with 200-, 400- and 600-grit silicon carbide paper with an automatic polishing machine (Grin PO 2V, Metkon A.Ş., Bursa, Turkey). Before the test procedure flexural test specimens were stored in a distilled water at 37 ºC for 50 ± 2 h and impact test specimens were stored at 37 ºC for 2 weeks for full saturation.

Three-point bending test was carried out using an universal testing machine (Lloyd, LRX, Fareham Hant, UK) with a crosshead speed of 5 mm/min (Figure 1). Specimens were placed to the jigs that were 50 mm apart and the force was applied through the center of specimens until a fracture occurs. Flexural strength (Fs) was calculated by the equation of:

$$FS=3FL/2bh^2$$

and the elastic modulus (E) calculated by:

$$E= Fl^3/4bh^3d$$

where F is loaded force, I is the distance between supports (50 mm), b is the width of the specimen (10 mm), h is the height of the specimen (2.5 mm) and d is the deflection (mm).

The impact test was conducted by the Charpy type impact tester (Ceast Resil Impactor, Italy) (Figure 2). Impact strength (IS) was calculated by the equation of:

$$IS = E/WT$$

where E is the energy that breaks the specimen (J), W is the width (6 mm) and T is the thickness of the specimen (4 mm).

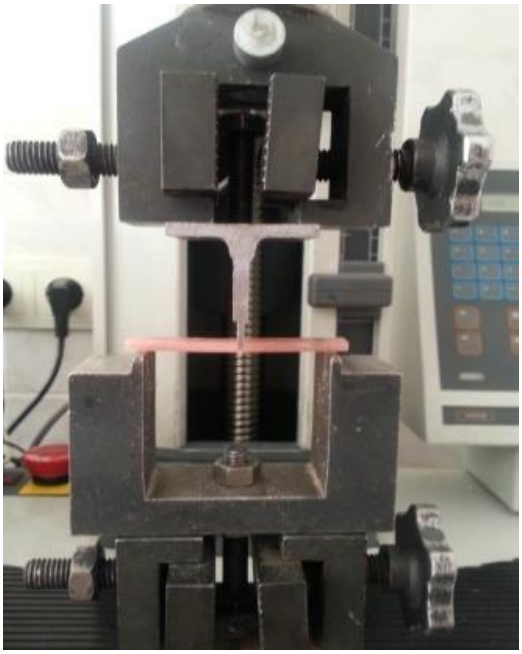

Figure 1.

Three -point bending test

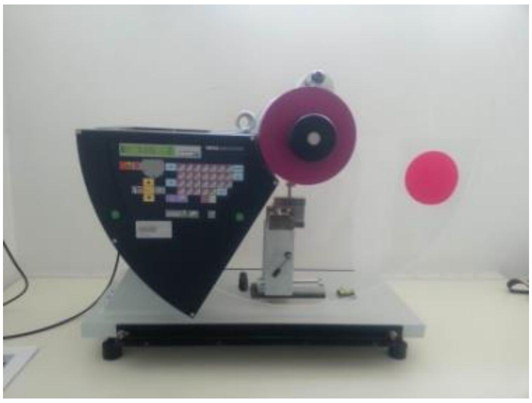

Figure 2.

Charpy type impact tester used in the study

For residual monomer extraction, test specimens were weighed accurately and each one placed in a glass flask containing 50 mL methanol. Glass flasks were connected to the Soxhlet extractor and placed in a water bath. The water bath's temperature was adjusted to 65 ºC considering the boiling point of methanol which is 64.5 ºC. Each glass flask containing specimen and methanol was kept in water bath for 6 h to remove residual monomer. To determine the residual monomer content high performance liquid chromatography (HPLC) (Thermo Scientific Dionex Ultimate 3000, Bremen, Germany) was used. Analysis was performed using Acclaim 120 C18 column, binary pump and auto injector system. Methanol-water isocratic mixture (70:30) was used as the mobile phase. Pump flow rate and injection volume were 1mL/min and 20 μL, respectively. Standard stock solutions of monomers were prepared by dissolving the monomers in methanol and different concentrations of stock solutions were injected into the HPLC system. By this way, obtaining the standard peaks of monomers, peak areas are recorded and measured to obtain calibration curve. The extract solution for each specimen was injected into HPLC system and residual monomer peak areas were obtained by using standard solution peak height and time localization. Afterwards, each extract's residual monomer fraction (v/v %) was determined and weight fractions of residual monomers were calculated for each sample.

Data were statistically analyzed, and computations were performed using statistical soft ware (SPSS v17.0; IBM Corp). The Levene's test of homogeneity was used to evaluate the distribution of the variables. The flexural strength, elastic modulus and impact strength results were separately analyzed with the three-way ANOVA test to evaluate the effects of resin material, added monomer type, added monomer ratio and their interactions. The mean flexural strength, elastic modulus, impact strength and residual monomer values were then compared with the Tukey HSD test ($\alpha=0.05$ for all tests).

RESULTS

According to the three-way ANOVA of flexural strength, elastic modulus, impact strength and residual monomer esults, the effect of the acrylic resin material, added monomer type and monomer ratio were statistically significant ($p<0.05$).

For flexural strength the interaction between resin material and monomer ratio and also the interaction between monomer type and monomer ratio were significant ($p<0.05$). Mean flexural strength values and standard deviations were shown in Table 2.

Table 2.

The means and standard deviations (SD) of the flexural strength values for test groups

Groups	Conventional Heat Polymerized Mean±SD (MPa)	Microwave Polymerized Mean±SD (MPa)
Control	94.24±3.11^{a}	178.29±12.73$^{\text{f-ı}}$
2% IBMA	115.66±7.77^{ab}	210.48±14.63$^{\text{hı}}$
5% IBMA	120.44±8.10$^{\text{a-d}}$	196.89±13.83$^{\text{g-ı}}$
10% IBMA	162.98±10.30$^{\text{d-g}}$	204.25±8.76$^{\text{g-ı}}$
2% BMA	115.81±8.69^{ab}	188.54±12.06$^{\text{f-ı}}$
5% BMA	123.54±8.13$^{\text{a-d}}$	187.99±13.64$^{\text{f-ı}}$
10% BMA	129.16±8.72$^{\text{a-e}}$	170.06±6.99$^{\text{e-h}}$
2% HEMA	109.08±10.40^{ab}	197.17±10.69$^{\text{g-ı}}$
5% HEMA	117.77±6.66$^{\text{a-d}}$	214.16±13.17$^{\text{hı}}$
10% HEMA	148.38±8.60$^{\text{b-f}}$	210.43±13.68$^{\text{hı}}$
2% POSS-MA	100.05±10.24^{a}	179.09±12.25$^{\text{f-ı}}$
5% POSS-MA	114.07±11.97^{ab}	219.42±13.75$^{\text{ı}}$
10% POSS-MA	116.96±7.72$^{\text{a-c}}$	161.196±11.45$^{\text{e-g}}$

**Results of Tukey post-hoc comparisons were shown as superscripts and values having same letters are not significantly different (p>0.05).*

When HP resin groups were evaluated, 10% IBMA group showed the highest (162.98 MPa) and control group showed the lowest (94.24 MPa) flexural strength values. There were significant differences between the control and 10% IBMA groups and 10% HEMA as well ($p<0.05$). For MP resins, 5% POSS-MA group was showed the highest (219.42 MPa) and 10% POSS-MA group showed the lowest (161.20 MPa) flexural strength values. However there was no significant difference between the control and copolymer groups ($p>0.05$).

For elastic modulus the interaction between resin material and monomer ratio and also the interaction between resin material and monomer ratio were significant ($p<0.05$). Mean elastic modulus values and standard deviations were shown in Table 3. When HP resin groups were evaluated, 10% IBMA and 10% HEMA groups showed the highest (2.49 GPa) and 2% POSS-MA showed the lowest (1.61 GPa) elastic modulus values. There was no significant differences between the control and copolymer groups ($p>0.05$). For MP resins, 5% POSS-MA groups showed the highest (4.26 GPa), control group showed the lowest (2.85 GPa) elastic modulus values. The addition of copolymers were usually resulted with an improvement on elastic modulus values but significant increases observed only for 2% IBMA, 5% IBMA, 5% HEMA, 10% HEMA and 5% POSS-MA groups ($p<0.05$).

Table 3.

The means and standard deviations (SD) of the elastic modulus values for test groups

Groups	Conventional Heat Polymerized Mean±SD (GPa)	Microwave Polymerized Mean±SD (GPa)
Control	1.72±0.10^{a}	2.85±0.40$^{\text{b-e}}$
2% IBMA	1.72±0.16^{a}	4.20±0.68^{h}
5% IBMA	1.71±0.40^{a}	3.91±0.46$^{\text{f-h}}$
10% IBMA	2.49±0.27$^{\text{a-d}}$	3.67±0.50$^{\text{e-h}}$
2% BMA	1.71±0.28^{a}	2.46±0.61$^{\text{a-d}}$
5% BMA	1.90±0.51^{ab}	3.03±0.66$^{\text{c-g}}$
10% BMA	2.16±0.51$^{\text{a-c}}$	2.93±0.35$^{\text{c-f}}$
2% HEMA	1.72±0.24^{a}	3.78±0.45$^{\text{e-h}}$
5% HEMA	1.89±0.12^{ab}	3.96±0.57^{gh}
10% HEMA	2.49±0.29$^{\text{a-d}}$	4.17±0.40^{h}
2% POSS-MA	1.61±0.29^{a}	3.38±0.45$^{\text{d-h}}$
5% POSS-MA	1.63±0.33^{a}	4.26±0.31^{h}
10% POSS-MA	1.78±0.14^{a}	3.54±0.62$^{\text{e-h}}$

**Results of Tukey post-hoc comparisons were shown as superscripts and values having same letters are not significantly different (p>0.05).*

For impact strength, the interaction between resin material and monomer type, the interaction between monomer type and monomer ratio also the triple interaction between resin type, monomer ratio and monomer type were significant ($p<0.05$). Mean impact strength values and standard deviations were shown in Table 4. When HP resin groups were evaluated 10% IBMA showed the highest (14.71 kJ/m^2) and 10% BMA showed the lowest (11.27 kJ/m^2) impact strength values. There was a significant difference between the control group and 5% POSS-MA group ($p<0.05$). For MP resin 2% HEMA group showed the highest (11.69 kJ/m^2) and 10% POSS-MA group showed the lowest (7.43 kJ/m^2) impact strength values. There was also a significant difference determined between the control and 10% POSS-MA groups ($p<0.05$).

Table 4.

The means and standard deviations (SD) of the impact strength values for test groups

Groups	Conventional Heat Polymerized Mean±SD (kJ/m^2)	Microwave Polymerized Mean±SD (kJ/m^2)
Control	13.05±1.76^{fg}	11.09±0.86^{b-f}
2% IBMA	13.02±2.05^{e-g}	8.90±1.24^{ab}
5% IBMA	13.64±1.33^{fg}	8.70±0.66^{ab}
10% IBMA	14.71±0.55^{g}	9.74±0.50^{a-e}
2% BMA	13.05±2.00^{fg}	11.24±1.23^{b-f}
5% BMA	12.97±3.01^{e-g}	9.38±1.22^{a-c}
10% BMA	11.27±1.69^{b-f}	8.90±1.12^{ab}
2% HEMA	12.80±1.74^{d-g}	11.69±1.26^{b-g}
5% HEMA	13.27±1.31^{fg}	9.62±0.57^{a-d}
10% HEMA	12.29±1.17^{c-g}	9.45±0.80^{a-c}
2% POSS-MA	11.80±0.90^{b-g}	8.79±1.76^{ab}
5% POSS-MA	9.15±0.67^{a-c}	10.43±0.87^{a-f}
10% POSS-MA	10.77±1.39^{b-f}	7.43±0.99^{a}

**Results of Tukey post-hoc comparisons were shown as superscripts and values having same letters are not significantly different (p>0.05).*

According to three-way ANOVA of residual monomer results, the interaction between resin material and monomer type, the interaction between monomer type and monomer ratio, the interaction between resin material and monomer ratio and also the triple interaction between resin type, monomer ratio and monomer type were significant (p<0.05). Table 5 summarizes the results of the mean residual monomer values and standard deviations.

When HP resin groups were evaluated 10 % BMA showed the highest (1.444 %w) and 2% POSS-MA showed the lowest (0.248 %w) residual monomer values. There were significant differences between the control group and 10% IBMA, 10%BMA and 10% POSS-MA copolymer groups (p<0.05). For MP resin 5% HEMA group showed the highest (0.349 %w) and control group showed the lowest (0.121 %w) residual monomer values. There were also significant differences determined between the control and 5% IBMA, 2% HEMA, 5% HEMA and 10% HEMA groups (p<0.05).

Table 5.

The means and standard deviations (SD) of the residual monomer values for test groups.

Groups	Conventional Heat Polymerized Mean±SD (weight %)	Microwave Polymerized Mean±SD (weight %)
Control	0.304±0.044^{e-h}	0.121±0.020^{a}
2% IBMA	0.326±0.013^{gh}	0.195±0.023^{a-d}
5% IBMA	0.349±0.027^{h}	0.218±0.020^{b-f}
10% IBMA	0.452±0.024^{ıj}	0.211±0.031^{a-e}
2% BMA	0.323±0.028^{gh}	0.149±0.014^{ab}
5% BMA	0.356±0.008^{hı}	0.149±0.006^{ab}
10% BMA	1.444±0.164^{k}	0.158±0.015^{a-c}
2% HEMA	0.340±0.013^{gh}	0.314±0.024^{f-h}
5% HEMA	0.323±0.015^{gh}	0.349±0.051^{h}
10% HEMA	0.345±0.026^{gh}	0.338±0.022^{gh}
2% POSS-MA	0.248±0.019^{c-g}	0.168±0.018^{a-c}
5% POSS-MA	0.265±0.029^{d-h}	0.186±0.025^{a-d}
10% POSS-MA	0.512±0.020^{j}	0.200±0.010^{a-d}

**Results of Tukey post-hoc comparisons were shown as superscripts and values having same letters are not significantly different (p>0.05).*

DISCUSSION

One of the approach to improve the mechanical properties of PMMA denture base materials is the addition of different monomers.[3,6,18,19] The aim of the present study was to obtain copolymer resin material by adding four different monomers to PMMA, and changing the matrix structure chemically. Also it was targeted to achieve an improved resin material having both hard and soft segments within the same molecule.[6,20]

In the present study flexural strength results showed that in HP resin groups compared to control group higher flexural strength values obtained by adding increasing amount of monomers. However only the addition of 10% IBMA and 10% HEMA were significantly improved the flexural strength of denture base resin. Strong Van der Walls attraction between MMA-IBMA and MMA-HEMA may be the reason of this situation.[6] The results of present study are consistent with the study findings of Şahin *et al*[6] which reported higher flexural strength values by the addition of 2 %, 3 % and 5% IBMA and HEMA into conventional PMMA denture base resin. POSS is a nanoscale organic-inorganic hybrid nanocomposite that have been developed at the end of the last century and represented with a formula of $RSiO_{1.5}$ where R may be a hydrogen atom or an organic functional group, e.g., alkyl, alkylene, acrylate, hydroxil or epoxide unit.[10,21-23] In several studies POSS have been used to improve the performance of the dental restorative materials by obtaining POSS -modified polymers.[10,21,24] Hamza *et al*[10] observed the effect of 1% POSS on the flexural strength of autopolymerizing PMMA resin and reported that no statistically significant difference was found. In the current study the addition of POSS-MA monomer had no significant effect in the flexural strength of both resin groups. This situation may be attributed to the study findings of Gao *et al*[25] which showed that miscibility between the POSS and the matrix played a very important role in improving the properties of resins.

In the current study the flexural strength values of microwave polymerized resin groups were higher than the conventional polymerized ones. Also for each copolymer group, there was no statistically significant difference between the control group and the copolymer groups. Compared to conventional water-bath method, in microwave polymerization, the curing procedure is much faster, the increase of the temperature is rapid and the heat inside and outside the substance is almost equal.[26,27] Therefore in the present study, due to the higher reaction rate, degree of conversion and internal heat produced during microwave irradiation, addition of copolymer didn't produce a positive effect on flexural strength for MP resin groups.[28]

Clarke[29] reported that in the main chain of PMMA by replacing the CH3- group with an alkyl group, that have higher numbers of carbon, the flexibility of the resin could be improved. In the current study in conventional polymerized resin group, similiar to the study findings of Şahin *et al*[6] the copolymerization reaction had no significant effect on the elastic modulus values. However, in MP copolymer resin groups, the higher elastic modulus values obtained after copolymerization.

Impact strength is the amount of energy which is absorbed by the material before failure. High impact strength materials easily absorbe the energy because of their elastic behavior. PMMA denture base is a hard and brittle material in the physiological temperature of oral cavity.[30] By adding rubber modifiers such as butadiene styrene the impact strength of the resin could be increased.[7] In the present study 5%POSS-MA added HP and 10% POSS-MA added MP resin groups had lower impact strength values compared to control groups. The other copolymer groups showed no statistically significant difference in terms of impact strength. In the mechanical properties of POSS-polymer composites the amount of POSS presents an important role. Wu *et al*[21] reported that by the addition of POSS above 10%, the mechanical properties of nanocomposites was found to deteriorated rapidly and this situation was explained by the rapid formation of cross-linked polymeric network with increasing POSS that limits the mobility of reactive species.

Residual monomer that leachs out from acrylic denture bases can cause allergic reactions, irritation and inflammation of oral tissues. Also, the residual monomer that acts as a plasticizer causes porosity and may affect the physical and mechanical properties of the resin.[12,31] Doğan *et al*[12] and Kedjarune *et al*[32] reported that the amount of monomer in the mixture, polymerization type and time effects the amount of residual monomer. In the current study the control group of MP resin indicated less residual monomer content compared to the control group of HP resin group. This situation is related to the microwave method in which high internal heat provided by the monomer molecules positively moved by a high – frequency electromagnetic field.[31] Also this result is in agreement with the study findings of Çelebi *et al*[31]

Copolymer type, amount and the effect of crosslinking agent within the polymer have a significant impact on many features of resins. In the present study, adding10 % IBMA, 10% BMA and 10% POSS-MA to the MMA of HP resin, significantly increased the residual monomer contents. HEMA is a water-soluble monomer and polimerizes at lower degrees. The presence of hydroxyethyl groups in polymer structure provides a hydrophilic character to the material.[33] In MP resin group by adding HEMA monomer to MMA, significant differences determined between the control and all HEMA copolymer groups.

This *in vitro* study has some limitations. Different monomer type and concentrations, long-term usage in clinical conditions and bacterial or candidal adhesion subjects should be taken into consideration for future investigations.

CONCLUSION

Within the limitations of this study it can be concluded that to obtain more durable copolymer denture base resins that have less residual monomer content, the addition of monomers should be in low concentrations.

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