Effects of water boiling, microwave, and water bath post-polymerization on mechanical properties of acrylic denture resins

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Abstract

Acrylic restorations in the mouth are exposed to strong occlusal forces. Their mechanical properties depend on the type and method of their polymerization. The aim of this study is a comparative analysis of mechanical properties (flexural strength and modulus of elasticity) of acrylic materials before and after the post-polymerization treatments (water boiling, microwave irradiation and water storage). The study included denture base resins, as well as an acrylate for orthodontic appliances impregnated with aesthetic beads. Flexural strength, modulus of elasticity and the deflection were measured immediately after polymerization, after a hot and microwave post-polymerization and after immersion in a water bath at the temperature of 37 °C. The applied post-polymerization methods resulted in an increase in flexural strength and modulus of elasticity relative to the initial values for all tested materials. Being aware of the reduction in fracture risk of dental prosthesis after a proposed post-polymerization procedure, it could be used in all clinical situations where there is an increased risk of damage to dentures.

Keywords: *acrylate, flexural strength, flexural modulus, fracture risk*.

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1. INTRODUCTION

Acrylates are frequently used in dental practice as restoratives, liners or as denture base materials [1-2]. These materials are divided as cold, heat, microwaved and light polymerized based on the initiation type of the polymerization reaction.

Acrylic dentures are artificial structures which replace lost teeth and parts of the resorbed alveolar ridge transferring masticatory forces to the bone beneath the oral mucosa. Acrylic orthodontic appliances with dosed forces move teeth in children leading them to an optimal dentoalveolar relationship. Frequent fractures of dentures and orthodontic appliances, caused by masticatory forces or by an accidental break due to inappropriate operation, are reasons for constant striving for improvement of mechanical properties of acrylic materials.

Good mechanical properties are one of the most important prerequisites for successful usage of acrylic resins in dentistry. Acrylates are not mechanically perfect materials. Even when they are optimally polymerized, their hardness, strength and impact resistance are lower compared to hard dental tissues. Small modulus of elasticity makes them brittle and prone to breaking [3-4]. On the other hand, dentures and orthodontic appliances in the mouth are exposed to repeated flexural forces. Midline fractures are related to the flexural strength of the resins, and this property is challenged every time the appliance undergoes cyclic functional deformation [5].

Mechanical properties vary within different acrylic material groups depending on differences in chemical structure of the materials, as well as the method and the length of the polymerization process [6-8]. Incomplete polymerization and monomer left in the structure of acrylate adversely affect the mechanical properties of the material [4]. The presence of residual monomer indicates formation of the polymer with lower molecular weight and porous structure, which significantly reduces the quality of acrylic restorations, hardness, tensile strength and modulus of elasticity and increases the absorption and solubility of liquids [9-15].

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Some studies have demonstrated that postpolymerization of acrylic denture resins and water storage can improve properties of acrylic materials [16-21]. Previous studies of Vergani *et al.* [16], Urban *et al.* [14] and Ozkomur and Fortes [19] showed positive impacts of additional polymerization procedures (microwave post-polymerization and immersion in boiling water) on the level of conversion of residual monomer in the acrylic polymer, and consequently the improvement in mechanical properties. Post-polymerisation treatment is especially recommended for cold polymerised acrylates, due to higher level of residual monomer after polymerization. Test methods to investigate some of the parameters are described by the ISO standard 4049 for polymer-based filling restorative and luting materials [22].

The aim of this study was a comparative analysis of the changes in mechanical properties (flexural strength and modulus of elasticity) of the acrylic materials before and after post-polymerization treatments (water boiling, microwave irradiation and water storage).

2. EXPERIMENTAL

Two hot and two cold polymerized acrylic based resins were selected for testing in this study. The resin characteristics, producers and polymerization methods are listed in Table 1. The study also included Ortopoli (OP) material impregnated with glittering particles (sequins) (OI), which are used in practice for aesthetics of orthodontic mobile dentures.

Table 1. Tested acrylic based resins

Product	Code	Manufacturer	Powder/Liquid ratio, g / ml	Composition		Polymerization condition
				polymer	monomer	
Triplex Hot	TH	IvoclarVivadent, Lichtenstein	2.34	PMMA	MMA EGDMA	45 min in boiling water
Lucitone 199	L	Dentsply International Inc. USA	2.1	PMMA	MMA	90 min at 72°C + 30 min in boiling water
Triplex Cold	TC	IvoclarVivadent, Lichtenstein	1.3	PMMA	MMA EGDMA	In pressure device at 40°C, 4 bar, 15 min
Ortopoli	OP	PoliDent, Slovenia	Spray-on technique	PMMA	MMA EGDMA	In pressure device at 40°C, 6 bar, 15 min

PMMA-Poly(methyl methacrylate); MMA- Methyl methacrylate; EGDMA-Ethylene glycol dimethacrylate

The aluminium rectangular-shaped patterns, 50×15×2 mm^3 were invested in denture flasks with a dental plaster. The flasks were opened and the patterns were removed, after setting of the plaster leaving cavities that were used as matrices for the fabrication of acrylic resin specimens. After mixing the acrylic resin according to the manufacturer's instructions, the molds were filled with the material.

After the standard polymerization procedure, the acrylic samples were taken out from the flasks. Excess of the material was eliminated by trimming the edges by using a handpiece at low speed, followed by additional hand smoothing by using a 320-grit silicon carbide paper.

The study included determination of the following mechanical properties: flexural strength and modulus of elasticity of the materials immediately after polymerization and after post-polymerization treatments. Post-polymerization treatment of the samples consisted of heat and microwave additional polymerization and water storage. The process and duration of the additional polymerization are explained in Table 2. For each tested material, 30 specimens were fabricated. There were six tested groups of the samples and each group consisted of 5 specimens.

Table 2. Post-polymerization treatments

Code	Tested group	Postpolymerization treatment	Duration of treatment
0	Control	No treatment	-
HP	Heat post-polymerization	Additional boiling in water inside the flask	30 minutes
MW	Microwave post-polymerization	Direct exposure to microwave radiation (650 W/3 min in microwave oven)[15]	3 minutes
WS1d	1 day storage	Storage in water bath at 37 °C (GFS, Germany)	1 day
WS7d	7 day storage		7 days
WS30d	30 day storage		30 days

Before testing, the width and thickness of each specimen were measured by a digital caliper (Mitutoyo, Japan) with measuring accuracy of ±0.1 mm. Only the resin specimens with slight variations in size (<0.3 mm) were included in the study, while the ones that were discarded were replaced by new ones.

The flexural strength and flexural modulus of the specimens were determined by using a three-point bending test device in a universal testing machine (Metro com, Italy). A tool for bending test consisted of two parallel stainless steel rollers (Ø 5 mm) that supported the specimen. The load was applied centrally by the third roller (Ø 5 mm). The specimens were cantered on the device in such a way that the loading wedge, set to travel at a crosshead speed of 5 mm/min, engaged the centre of the upper surface of the specimens. The measurement was terminated at the sample fracture.

The span of this three-point deflection test was 30 mm. Force measurements were performed by a load cell HBM U2B/500 N (Germany) while deflection by a displacement transducer (HBM WA/10 mm, Germany). Recording of the measuring results was performed by using a measure unit DMC 9012 (Germany) until the specimen fracture. The maximum force [N] upon fracture was recorded.

The flexural strength (R_{bm}) was calculated by the equation (1) [8,23]:

$$R_{bm} = \frac{3Fl}{2wt^2} \quad (1)$$

where: R_{bm} - bending/flexural strength, F - maximum load exerted on the specimen, l - distance between the supports, w - width of the specimen, t - thickness of the specimen.

Results of flexural strength tests are used to calculate flexural moduli. Additionally, the specimen deflection and the corresponding force were determined. The flexural modulus (E) was calculated by the equation (2) [5,8]:

$$E = \frac{F_l l^3}{4wt^3 d} \quad (2)$$

where: F_l - the load at a convenient point in the straight-line portion of the trace and d- the deflection at the load F_l.

2. 1. Statistical analysis

All statistical analyses were performed using the IBM SPSS Statistics version 15.0. The statistical significance of differences of continuous variables after performing the procedures (HP and MW) in comparison to the initial values was determined by Paired samples t-test (normal distribution of variables) or Wilcoxon signed rank test (distribution of variables deviates from the normal distribution). The distribution normality of values of continuous variables has been determined by the Shapiro-Wilk test of normality. Statistical significance was defined as a p value < 0.05.

3. RESULTS AND DISCUSSION

Applied post-polymerization methods resulted in an increase in flexural strength as compared to the initial value for all tested materials.

After heat post-polymerization, a statistically significant increase in flexural strength was achieved only in the case of Triplex Cold ($p < 0.05$), while the microwave post-polymerization produced statistical significances for both investigated cold polymerized acrylates and Triplex Hot ($p < 0.01$). Prolonging the WS duration induced the increase in flexural strengths as compared to the initial values for all tested materials. A statistically significant increase was achieved after one-day of WS for Triplex Cold ($p < 0.05$), after 7 days for Triplex Cold and Ortopoli ($p < 0.05$), but after 30 days for Triplex Hot and Ortopoli ($p < 0.05$) and Triplex Cold ($p < 0.01$) (Table 3).

Compared to the initial values, there was an increase in modulus of elasticity for all tested materials after HP, MW and WS.

After MW, a statistically significant increase was achieved in the case of Triplex Cold ($p < 0.05$), and in particular in Triplex Hot ($p < 0.01$). Modulus of elasticity increased with the WS duration. However, a statistically significant increase was recorded only for Triplex Hot after 7 and 30 days ($p < 0.05$) (Table 4).

Applications of HP and WS have not induced significant changes in deflection of materials at 10 N/mm^2 as compared to the initial values. Deflection reduction at 10 N/mm^2 was noticed in all materials after one-day, seven-day and thirty-day WS, being all the more prominent with increasing duration of WS. However, a statistically significant decrease ($p < 0.05$) was found in Ortopoli with impregnation samples, only, after thirty-day WS (Table 5).

Deflection value at 25 N/mm^2 in all materials decreased after HP and WS as compared to the initial values, but these changes were not statistically significant. Deflection reduction at 25 N/mm^2 increased proportionally with the increase in WS duration. A statistically significant decrease after 30 days of WS ($p < 0.01$) was found for Lucitone, Ortopoli and Ortopoli with impregnation samples as compared to the initial values (Table 6).

Table 3. *Values of flexural strengths: initial, after HP, after MW and WS (1, 7 and 30 days)*

Material	Initial			After HP			After MW		
	R_{bm} / MP*a*	SD	Median	R_{bm} / MPa	SD	Median	R_{bm} / MPa	SD	Median
Triplex Hot	110.25	7.68	113.00	115.50	4.36	116.50	143.50	3.70	143.00**
Triplex Cold	99.25	8.73	98.00	124.75	5.85	124.50*	128.00	5.89	129.00*
Lucitone	113.50	13.63	111.50	121.50	3.11	121.50	131.75	10.87	128.50
Ortopoli	84.75	8.85	87.50	109.75	8.18	109.50	112.00	4.08	112.00**
Ortopoli with impregnation	85.25	2.87	86.50	106.00	18.02	105.00	103.00	12.52	104.50
	WS1d			WS7d			WS30d		
	R_{bm} / MPa	SD	Median	R_{bm} / MPa	SD	Median	R_{bm} / MPa	SD	Median
Triplex Hot	114.50	16.62	108.50	120.00	6.48	120.50	126.25	5.97	124.00*
Triplex Cold	120.25	2.22	120.00*	122.75	3.86	122.00*	126.00	5.29	127.00**
Lucitone	115.50	5.07	113.50	118.50	3.42	118.00	129.25	0.96	129.50
Ortopoli	86.75	7.93	88.00	105.00	4.97	105.50*	106.00	4.24	106.50*
Ortopoli with impregnation	86.75	5.91	85.50	99.50	9.47	98.50	103.25	5.56	103.00

*p < 0.05, **p < 0.01; (Paired samples t-test or Wilcoxon signed rank test); *SD-Standard Deviation*

Table 4. *Values of flexural moduli: initial, after HP, after MW and WS (1, 7 and 30 days)*

Material	Initial			After HP			After MW		
	E/ MPa	SD	Median	*E*/ MPa	SD	Median	*E*/ MPa	SD	Median
Triplex Hot	1399.75	8.18	1399.50	1424.50	25.05	1429.0)	1548.50	52.40	1555.00**
Triplex Cold	1255.00	12.91	1255.00	1415.50	43.22	1396.00	1410.00	54.77	1390.00*
Lucitone	1387.50	106.85	1425.0)	1422.00	107.34	1414.00	1426.25	115.86	1415.00
Ortopoli	1237.50	72.74	1235.00	1247.50	73.65	1275.00	1250.00	163.91	1295.00
Ortopoli with impregnation	1227.50	28.72	1230.00	1247.50	49.92	1255.00	1245.00	131.78	1295.00
	WS1d			WS7d			WS30d		
	E/ MPa	SD	Median	*E*/ MPa	SD	Median	*E*/ MPa	SD	Median
Triplex Hot	1451.25	64.86	1445.00	1485.00	41.23	1480.00*	1577.50	76.32	1595.00*
Triplex Cold	1300.25	77.66	1327.50	1374.50	98.21	1354.00	1575.00	43.59	1595.00
Lucitone	1407.50	56.79	1385.00	1415.00	68.07	1430.00	1517.50	59.65	1540.00
Ortopoli	1255.00	72.34	1245.00	1285.00	78.53	1265.00	1297.50	103.40	1310.00
Ortopoli with impregnation	1230.00	62.18	1250.00	1263.75	86.35	1272.50	1282.50	23.63	1290.00

*p < 0.05, **p < 0.01 (Paired samples t-test or Wilcoxon signed rank test)

Acrylic materials largely satisfy requirements defined during the long clinical practice. Acrylate restorations exposed to severe occlusal forces in the patient's mouth exhibit adequate mechanical properties such as modulus of elasticity, flexural strength, micro-hardness, and impact strength, in order to be integrated in the orofacial system [24,25].

Some studies have shown that additional polymerization, hot and microwave post-polymerization can improve mechanical properties of these materials [17,18]. Results from the present study demonstrated the effects of water boiling, microwave and water bath post-polymerization treatments on mechanical properties of two heat polymerized and two cold polymerized denture base resin materials. After post-polymerization procedures, there was an increase in the value of flexural strength as well as in modulus of elasticity in all tested materials compared to the values obtained immediately after material preparation. The measured values increased with the duration of immersion in a water bath.

Table 5. *Values of deflection at 10 N/mm²: initial, after HP, after MW and WS (1, 7 and 30 days)*

Material	Initial			After HP			After MW		
	d / mm	SD	Median	*d* / mm	SD	Median	*d* / mm	SD	Median
Triplex Hot	0.18	0.03	0.18	0.17	0.01	0.17	0.17	0.02	0.17
Triplex Cold	0.19	0.05	0.19	0.19	0.06	0.19	0.20	0.02	0.19
Lucitone	0.24	0.02	0.24	0.25	0.03	0.25	0.26	0.02	0.27
Ortopoli	0.30	0.06	0.32	0.30	0.03	0.31	0.29	0.03	0.30
Ortopoli with impregnation	0.26	0.03	0.25	0.26	0.05	0.25	0.25	0.03	0.26
	WS1d			WS7d			WS30d		
	d / mm	SD	Median	*d* / mm	SD	Median	*d* / mm	SD	Median
Triplex Hot	0.17	0.02	0.17	0.16	0.01	0.16	0.13	0.01	0.13
Triplex Cold	0.18	0.02	0.18	0.16	0.02	0.16	0.13	0.01	0.14
Lucitone	0.23	0.02	0.23	0.22	0.02	0.23	0.15	0.06	0.13
Ortopoli	0.27	0.03	0.27	0.26	0.05	0.25	0.21	0.06	0.20
Ortopoli with impregnation	0.24	0.01	0.25	0.23	0.04	0.24	0.17	0.00	0.17*

*p < 0.05, (Paired samples t-test or Wilcoxon signed rank test)

Table 6. Values of deflection at 25 N/mm²: initial, after HP, after MW and WS (1, 7 and 30 days)

Material	Initial			After HP			After MW		
	d / mm	SD	Median	*d* / mm	SD	Median	*d* / mm	SD	Median
Triplex Hot	0.47	0.02	0.46	0.46	0.03	0.46	0.46	0.03	0.47
Triplex Cold	0.33	0.02	0.33	0.31	0.03	0.32	0.31	0.08	0.32
Lucitone	0.67	0.09	0.68	0.64	0.02	0.64	0.66	0.08	0.64
Ortopoli	0.70	0.08	0.72	0.70	0.04	0.69	0.64	0.04	0.64
Ortopoli with impregnation	0.67	0.05	0.69	0.66	0.09	0.66	0.64	0.13	0.67
	WS1d			WS7d			WS30d		
	d / mm	SD	Median	*d* / mm	SD	Median	*d* / mm	SD	Median
Triplex Hot	0.45	0.09	0.45	0.40	0.13	0.40	0.34	0.02	0.35
Triplex Cold	0.32	0.01	0.33	0.28	0.05	0.29	0.27	0.05	0.28
Lucitone	0.62	0.05	0.60	0.52	0.07	0.53	0.39	0.11	0.38**
Ortopoli	0.63	0.11	0.59	0.61	0.09	0.65	0.49	0.03	0.50**
Ortopoli with impregnation	0.65	0.02	0.66	0.63	0.14	0.68	0.48	0.01	0.48**

**p < 0.01, Paired Samples Test or Wilcoxon Signed Ranks Test)

These results can be explained by a decrease in the amount of residual monomer, thereby improving mechanical quality of the material. In specific, Kostic *et al.* showed a significant reduction in the amount of residual monomers in two hot and two cold polymerized acrylic materials after described post-polymerization procedures, by a HPLC method (high performance liquid chromatography)[20, 26].

In support of this theory is the fact that a statistically significant increase in the value of flexural strength was observed in cold-polymerized acrylates (Triplex Cold after HP, Triplex Cold and Ortopoli) after WS. Namely, cold polymerization is less complete than the one in boiling water, and there is a greater potential for additional binding of the residual monomer.

Flexural strength testing conducted in the present investigation is clinically relevant since it reflects the loading arrangement in the clinical situation [27]. Rigidity of the material is reflected by the flexural modulus, which in turn is important for the integrity of the supporting ridge and tissues, along with the fitting accuracy of the denture [28-30].

Denture base acrylate must not deform under loading in order to permit proper load distribution to the underlying structure [31].

Improvement in mechanical characteristics of base resins reduces the possibility of fracturing when the prosthesis is dropped onto a hard surface [31-35]. Therefore, if post-polymerization results in a significant increase in flexural strengths of acrylic resins, this may decrease the possibility of a fracture occurring inside or outside the oral cavity.

On the other hand, statistically significant increases in the modulus of elasticity after MW and seven-day immersion in water bath have been observed in heat polymerized Triplex Hot, improving its resistance to breakage.

HP and WS have not resulted in significant changes in deflection of materials at 10 N/mm^2 compared to the initial values although it slightly but not significantly decreased with the duration of WS. Similarly, deflection at 25 N/mm^2 decreased slightly but not significantly in all materials after HP and WS as compared to the initial values. A statistically significant decrease was achieved only after 30 days of WS for materials Lucitone, Ortopoli and Ortopoli with impregnation.

Generally, the polymerization cycle of heat-polymerized acrylic resins should include a terminal boiling treatment for at least 1 hour to achieve the maximal monomer conversion. Although the recommended polymerization cycle for Triplex Hot and Lucitone 199 included a terminal boil, the materials were maintained at 100 °C for 45 and 30 minutes, respectively.

The polymerization degree in polymerization initiated by a chemical activator is significantly lower as compared to that in hot polymerization [33-35]. Heating the cold polymerized samples in boiling water additionally binds residual monomers in the polymer matrix. Thomson and Luo concluded that mechanical properties of methacrylates can be improved by post-polymerization heat treatments [36]. Gad *et al.* [37] and Abdulwahhab [38] showed a significant increase in flexural strength and hardness of heat polymerized dental base resins upon post-polymerization treatment.

Previous studies have shown possibilities for usage of microwave post-polymerization to decrease the amount of residual monomer and thus increase the acrylic material quality. Vergani *et al.* [39], Urban *et al.* [40], and Blagojević and Murphy [17] noticed that the flexural strength of cold-polymerized denture base resins was positively affected by microwave post-polymerization. Ozkomur and Fortes demonstrated a significant increase in flexural strength after post-polymerization by microwave irradiation [19]. Patil *et al.* [41] and Takahashi *et al.* [42] concluded that post-polymerization by microwave irradiation can be an effective method for increasing the flexural strength of a denture acrylic liner (650 W for 5 min) due to reduction of the residual monomer content by further polymerization. Also, Araujo *et al*. reported that microwave electromagnetic radiation can be employed for monomer content reduction by additional polymerization [33].

In the present study, the materials were microwaved in a dry atmosphere since the water uptake by the acrylic resin would lead to resin plasticization, making it more flexible and resilient [43]. Therefore, diffusion and hydrolysis mechanisms were not involved in the reduction of the residual monomer content when microwave irradiation was used. As discussed for the boiling post-polymerization treatment, heating generated during microwave irradiation probably provided additional polymerization, thereby reducing the residual monomer content [42]. In addition, the monomer content decrease by microwave heating could be due to monomer volatilization as suggested elsewhere. Thus, the reduced amount of residual monomer is probably the result of a combination of the above mechanisms [44,45]. However, further studies are necessary to validate this assumption.

Post-polymerization in a water bath can be an alternative way to improve mechanical characteristics of acrylates, by diffusion of non-polymerized monomer into the water [46] and possibly by additional polymerization in places with active radicals [47,48]. Studies of Wolff et al. also point to the residual monomer release after immersion in water, proportional to immersion duration within the observation period [49].

Author's earlier studies have shown possibilities for decreasing the amount of residual monomer and other potentially toxic substances in acrylic material samples by additional polymerization and immersion in a water bath [20,50]. Kostic et al. investigated the effects of residual monomer reduction by immersion in a water bath on pressure yields stress and superficial structure of acrylic materials [26]. Increase in mechanical properties and homogenization of the surface was noticed in all tested samples [26]. Correspondingly, Messe has demonstrated higher hardness values of investigated denture liners after immersion in a water bath [51].

It should be noticed that a more complete polymerization resulted in the improvement of material mechanical properties, but also increased material biocompatibility, given the potentially toxic effects of unbound monomers [52,53].

4. CONCLUSION

Higher values of flexural strength and modulus of elasticity were observed in all tested hot and cold polymerized acrylates after a heat, microwave post-polymerization and immersion in a water bath. Furthermore, the measured values

increased with the duration of immersion in a water bath. Heat and microwave post-polymerization procedures resulted in statistically significant increases in flexural strength and modulus of elasticity in Triplex Cold samples ($p < 0.05$).

Bearing in mind reduction in risk of fracture of dental prosthesis after the proposed post-polymerization procedures, they can be used in all clinical situations with the increased risk of denture damage. This is particularly significant for frequently repaired dental restorations.

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SAŽETAK

UTICAJ KUVANJA U VODI, MIKROTALASNOG ZRAČENJA I DRŽANJA U VODENOM KUPATILU NA MEHANIČKE KARAKTERISTIKE AKRILATA

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(Naučni rad)

Nadoknade od akrilata u ustima su izložene snažnim okluzalnim silama. Njihove mahaničke karakteristike zavise od vrste i načina polimerizacije. Cilj ove studije je komparativna analiza mehaničkih karakteristika (otpornost na savijanje i modul elastičnosti) akrilata pre i nakon postpolimerizacionih tretmana (ključala voda, mikrotalasno zračenje i potapanje u vodi temperature 37 °C). Istraživanje je uključilo akrilate za baze proteze kao i akrilate za ortodontske namene impregnirane estetskim perlama. Otpornost na savijanje, modul elastičnosti i ugib mereni su odmah nakon polimerizacije i nakon različitih postpolimerizacionih tretmana. Primenjene postpolimerizacione metode dovele su do porasta otpornosti na savijanje i modula elastičnosti u odnosu na početne vrednosti za sve testirane materijale. Imajući u vidu smanjenje mogućnosti frakture zubnih proteza nakon predloženih postpolimerizacionih procedura, one se mogu koristiti u svim kliničkim situacijma gde postoji povećani rizik za oštećenje zubnih nadoknada.

Ključne reči: akrilat, savojna čvrstoća, modul elastičnosti, rizik za lom