

# Effect of Butadiene Acrylate Cross-Linking on Impact Strength of Poly Methyl Methacrylate

Učinak Butadin-akrilnog umreživača na udarnu čvrstoću poli(metilmetakrilata)

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## Summary

*A cross-linking agent, ethylene glycol dimethacrylate, which is added to poly (methyl methacrylate) (PMMA) denture base resins to improve crazing resistance, has little effect on the mechanical properties of the resultant denture base. A polyfunctional cross-linking agent, butadiene acrylate (BDA), has been considered to have a greater cross-linking efficiency and thus altering the mechanical properties. The purpose of this study was to investigate the effect of BDA on the impact strength of a PMMA specimens containing 0%, 1%, 4% and 12% BDA of monomer volume which had been produced by a conventional dental dough-moulded technique as determined. Mean impact strengths were 1.789 KJ/m<sup>2</sup> (SD 0.254), 1.720 KJ/m<sup>2</sup> (SD 0.273); 1.715 KJ/m<sup>2</sup> (SD 0.280) and 1.838 KJ/m<sup>2</sup> (SD 0.244), respectively. These differences were not significant ( $p > 0.05$ ) as tested by the analysis of variance. It was concluded that the cross-linking agent BDA did not affect the impact strength of the PMMA resin specimens.*

**Key words:** poly(methyl methacrylate), cross-linking, butadiene acrylate, impact strength

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## Introduction

The cross linking agent ethylene glycol dimethacrylate which is added to poly (methyl methacrylate) denture base resins to improve craze resistance, has little effect on the impact properties of the resultant denture base. It was considered that the poly functional cross linking

agent butadiene acrylate (BDA) might have greater cross linking efficiency and thus alter impact properties. The purpose of the present study was to investigate the effect of BDA on the impact strength of notched PMMA specimens produced by conventional dental dough moulded techniques containing BDA cross-linking agent (Chemoz Ltd, Esher, Surrey, UK) in

concentrations of 1%, 4% and 12% of monomer volume.

### Materials and Methods

The powder component used was unpigmented poly (methyl methacrylate) homopolymer (Bonar Polymers Ltd, Co Durham, UK). This powder was extensively characterised for a previous study and details have been published (1).

Table 1. *Characterisation of powder component*  
 Tablica 1. *Opis polimerskog praha*

Average Particle Size	64.85 $\mu\text{m}$
Weight Average Molecular Weight (Mw)	$1.59 \times 10^6$
Number Average Molecular Weight (Mn)	$4.82 \times 10^5$
Initiator Content (Benzoyl Peroxide)	0.25%

A summary of the characterisation is presented in Table 1. The liquid component was methyl methacrylate monomer stabilised with 0.006% hydroquinone (British Drug House Ltd, Poole, UK). The molecular structure of the cross linking agent BDA, which is added to the monomer component, is presented in Figure 1.

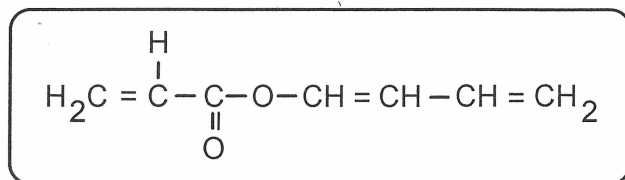


Figure 1. *Molecular structure of the cross-linking agent BDA (butadiene acrylate)*

Slika 1. *Molekularna struktura umreživača BDA (butadien-akrilat)*

### Test Specimen Preparation

Specimens were produced following carefully standardised methods of preparation. The moulds were prepared by investing master pattern blanks of the appropriate dimensions in gypsum using conventional dental flasking techniques. A powder: liquid ratio of 3.2:1 v/v was used to form a dough. Mould separation,

packing and clamping procedures followed standard practice. The materials were polymerised in a water bath. The clamped flasks were placed in cold water and the temperature raised to 70°C and maintained at that temperature for 7 hours, then temperature was further raised to 100°C and maintained at that temperature for 3 hours. The moulds were allowed to bench cool prior to devesting.

After devesting, the specimens were trimmed using progressively finer grades of silicon carbide grit paper, finishing with grade 600. Final dimensions of test specimens were 50 x 6 x 4 mm. All specimens were saturated in water for 28 days at 37°C prior to testing. The specimens were notched using a milling machine with multi tooth cutter (Adcock and Shipley, Zwick, T.M., Leominster, UK). The notch depth was 0.8 mm and notch tip radius 0.25 mm.

Fracturing of the specimens followed the procedure described in British Standard 2782 (1984) for the determination of Charpy impact strength of rigid materials.

The Zwick Pendulum Impact Tester (Zwick T. M. Leominster, Herts, U.K.) was used for testing the impact resistance. This is a Charpy type machine which utilizes a rectangular specimen bar that is supported at both ends, and struck by the pendulum in the centre directly behind the notch. It causes fracture of the specimen by means of a ballistic pendulum. The machine gives the energy absorbed by the specimen during fracture. All specimens were tested using a 0.5 Joule pendulum. Ten specimens of each group were fractured by releasing the pendulum from its highest position. The impact energy was displayed digitally. The results were expressed in KJ/m<sup>2</sup> after calculation of fracture surface areas.

### Results

The mean Zwick notched impact strengths of specimens containing 0%, 1%, 4% and 12% BDA of monomer volume are given in Table 2. Mean impact strengths were 1.789, 1.720, 1.715 and 1.838 KJ/m<sup>2</sup> respectively. To identify any significant differences between the data obtained a one-way analysis of variance was undertaken. The differences were not significant ( $p = 0.69$ ).

Table 2. Charpy (energy) strength values determined using Zwick apparatus ( $F = 0.49$ ;  $df = 3 - 36$ ;  $P = 0,69$ )Tablica 2. Charpy (energijske) vrijednosti čvrstoće dobivene Zwick-instrumentom ( $F = 0.49$ ;  $df = 3 - 36$ ;  $P = 0,69$ )

Percentage Cross-linking (BDA) v/v	Mean ( $n = 10$ ) KJ/m	S.D.	CofV %
0	1.789	0.254	14
1	1.720	0.273	16
4	1.715	0.280	16
12	1.838	0.244	13

## Discussion

The importance of impact testing of denture base materials has been acknowledged by many workers and Charpy and Izod test methods have been described in the dental literature (2 - 12).

The impact testing of polymeric materials is recognised as a difficult and controversial subject and a remarkably large number of impact test methods have been devised to study the impact behaviour of polymers. Specimens have been thrown, dropped and subjected to blows from hammers, bullets, falling weights, falling balls, horizontally moving balls, pendulums and projections from flywheels (13). Not surprisingly, with such diverse testing procedures, comparisons and results are of little value and it is accepted that no simple test can offer an unquestionable assessment of the impact resistance of any material.

The swinging pendulum-type impact tester has become the most often used for routine impact testing and of these there are two main categories:

(a) the Charpy test - where the specimen is supported horizontally at its end;

(b) the Izod test - where the specimen is clamped at one end held vertically. In both cases the resulting loss in swing amplitude after breaking the specimen is measured and converted into an impact energy. This is usually quoted as energy per unit dimension for Izod tests (joules per metre or foot pound force per inch), or in energy per unit cross section for Charpy tests (joules per metre squared or feet pounds foot per inch squared).

Notches are often intentionally put into Izod or Charpy test specimens to assess the so called

notch sensitivity of materials or, sometimes, just to cause the test specimens to fracture rather than just deform. The most critical characteristic of such notches is the notch tip radius, and it has been demonstrated that the impact energy of many polymeric materials can vary quite appreciably when the radius is changed over a practical range (0.25 - 2.5 mm); this can be a measure of notch sensitivity. A notch tip radius of 0.25 mm is often used as a standard; it corresponds to a sharp notch, and the associated severe stress concentration. Notches are placed in the most stressed section of Izod or Charpy test specimens and on the tensile side, since this is where the fracture begins.

A disadvantage of impact tests is that they do not measure a constant of the material. Energy is expended in initiating a crack, propagating it through the specimens and throwing the broken pieces of the specimen from the machine. In general, impact tests are only of value when brittle, rather than ductile, failure is studied. Poly (methyl methacrylate) is one of the most suitable thermoplastics for impact testing since its stress-strain relation does not depart very far from the assumptions of linear elastic theory and its impact energy is known to be unaffected by temperature changes in the room temperature region. Further, it is reported that a good correlation exists between Izod impact and Charpy impact test results for several thermoplastics under various test conditions (14).

In the dental literature, the impact testing of denture base polymers has been mainly concerned with PMMA and comparison with other denture base polymers. Polymers such as the polyamides, polycarbonates and rubber toughened acrylic resins are frequently shown to be superior regarding impact resistance.

In general, impact strength of a cross-linked polymer is reduced with increasing cross-linking concentration (15). Cornell et al. (7) found an improvement in impact resistance to falling balls when 10 or 20% EGDMA was used. This agreed with the study carried out by Price (16). In her study, the impact resistance of reversed notched specimens was increased with low concentrations of EGDMA (up to 10%), and decreased with higher concentrations. However, the impact resistance of notched specimens did not show any increase at all and decreased with increasing the concentration of EGDMA. On the other hand, Harrison, Jagger and Huggett

(17) found no significant effect on the impact resistance of notched specimens which were prepared to the dimensions which are given in the British Standard (BS 2782). This is probably due to the fact that materials cross-linked with EGDMA show high notch sensitivity with increasing concentration of cross-linking in the monomer (16).

In summary, EGDMA has little effect on mechanical properties of two-phase denture ba-

se materials except to reduce tensile strength and impact resistance when added to the monomer in high concentrations. However, the impact resistance and tensile strength increased with low percentage of EGDMA.

From this study it was concluded that BDA has no effect on the impact strength of PMMA dough moulded resin when added in up to 12% of monomer volume.

#### UČINAK BUTADIN-AKRILATNOG UMREŽIVAČA NA UDARNU ČVRSTOĆU POLI(METILMETAKRILATA)

##### Sažetak

*U svrhu povećanja otpornosti poli(metilmetakrilatnih) protez-nih smola na lomove i stvaranje napuklina, monomer-kapljevini se tvornički dodaje tvar za umrežavanje, najčešće etilen-glikol-dime-takrilat (EGDMA), koji se u tome smislu nije pokazao učinkovitim. Ovo istraživanje je provedeno s pretpostavkom da bi poli-funkcionalna tvar za umrežavanje, kao što je to butadin – akrilat (BDA), mogla imati veći učinak na umrežavanje i poboljšanje mehaničkih svojstava poli(metilmetakrilatnih) smola. Ispitivana je udarna čvrstoća Zwick-postupkom poli(metilmetakrilatnih) pri-pravaka, podijeljenih u četiri skupine s obzirom na količinu tvari za umrežavanje (0%, 1%, 4% i 12%), te dobivenih uobičajenom kivetnom tehnikom topopolimerizirajućeg postupka. Vrijednosti za udarnu čvrstoću pripravaka po skupinama iznosile su: 0% BDA – 1,789 KJ/m<sup>2</sup> (SD 0,254); 1% BDA – 1,720 KJ/m<sup>2</sup> (SD 0,273); 4% BDA – 1,715 KJ/m<sup>2</sup> (SD 0,280) i 12% BDA – 1,838 KJ/m<sup>2</sup> (SD 0,244), a razlike među skupinama nisu se pokazale statistički značajnim ( $p > 0,05$ ). Zaključeno je da istraživana tvar za umrežavanje (BDA) nije doprinijela poboljšanju udarne čvrstoće poli(metilmetakrilatnih) pripravaka.*

**Ključne riječi:** poli(metilmetakrilat), umrežavanje, butadin-akrilat, udarna čvrstoća

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