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Mechanical Properties of Poured Aramid Fiber Reinforced Acrylic Resin Depending on Fiber Quantity, Fiber Position and Different Wetting Agents

Właściwości mechaniczne wlewowej żywicy akrylowej wzmocnionej włóknem aramidowym w zależności od liczby i położenia włókien oraz różnych czynników wiążących

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Abstract

Background. Acrylic resin used in restorative dentistry is the most popular denture base material. However due to its limited mechanical properties, different methods to reinforce are required.

Objectives. To investigate the effect of various quantities and position of aramid fibers (AF) covered by different bifunctional acrylic monomers on the flexural properties of cold curing acrylic resin (AR) suitable for the pouring method.

Material and Methods. Colacryl TS 2027® (Lucite) denture base test specimens were reinforced with various quantity unidirectional aramid fibers Tawaron® (Teijin) in different position in the samples. The fibres surface was pretreated with methacrylic polymer solution alone and with combinations of methacrylic monomers (Bis GMA, UDMA, methacrylic oligomer with polycarboxylic acid group). Control specimens were not fiber reinforced. Flexural properties of aramid fibers reinforced acrylic resins (AFRAR) were determined in dry condition after 24 hours at 23° ± 1°C and after 7 day water immersion at 37° ± 1°C. The test was performed by a three-point bending test using Instron type 4411 tensile testing machine.

Results. Flexural resistance of the aramid fiber reinforced acrylic resin (AFRAR) grew with a increasing number of fiber bundle in the sample. Positioning of fibers in the opposite direction to the breaking force effectively increased the flexural strength. Best results were obtained with Bis GMA methacrylic monomer which is a good cross linking agent for AF.

Conclusions. Unidirectional AF with methacrylic polyfunctional monomers pretreated surface, may find application in restorative dentistry for the purpose of increasing the mechanical properties of poured acrylic resins used as denture base material (*Dent. Med. Probl.* 2011, 48, 3, 380–387).

Key words: aramid fibers, acrylic resin, mechanical properties.

Streszczenie

Wprowadzenie. Tworzywo akrylowe stosowane w stomatologii odtwórczej, jako najbardziej popularny materiał do wykonania protez ruchomych, wykazuje ograniczoną wytrzymałość mechaniczną i dlatego proponuje się różne metody jego wzmocnienia.

Cel pracy. Określenie wpływu liczby oraz położenia włókna aramidowego (AF) zwilżonego różnymi środkami łączącymi na właściwości mechaniczne wlewowej żywicy akrylowej (AR).

Materiał i metody. Próbkę z żywicy akrylowej Colacryl TS 2027® (Lucite) wzmocniono jednokierunkowym włóknem aramidowym Tawaron® (Teijin), które umieszczano w górnej i dolnej części próbek, wielokierunkowo oraz pionowo. Włókno było wcześniej zwilżone różnymi czynnikami łączącymi (tylko metakrylanem metylu oraz w kombinacji z innymi monomerami). Oceniono właściwości mechaniczne wzmocnionego tworzywa przechowywanego w warunkach suchych w temperaturze 23 ± 1°C przez 24 godz. po wykonaniu próbek oraz po 7 dniach przechowywania w wodzie w temperaturze 37 ± 1°C. Próbkami odniesienia były próbki niewzmocnione włóknem aramidowym. Badanie wykonano w zrywarcie Instron typu 4411.

Wyniki. Wytrzymałość na zginanie próbek akrylowych wzmocnionych włóknami aramidowymi (AFRAR) zwiększyła się ze zwiększeniem liczby włókien w próbce, a także wówczas, gdy włókno było ułożone po stronie przeciw-

nej do kierunku działania siły. Najkorzystniejsze wyniki otrzymano dla monomeru Bis GMA, który tworzy wiązania wodorowe z włóknami aramidowymi.

Wnioski. Jednokierunkowe włókna aramidowe o powierzchni zwilżonej wielofunkcyjnym monomerem metakrylowym mogą znaleźć zastosowanie w stomatologii odtwórczej w celu polepszenia mechanicznych właściwości wlewowych żywic akrylowych stosowanych do wykonania płyt protez (**Dent. Med. Probl.** 2011, 48, 3, 380–387).

Słowa kluczowe: włókna aramidowe, żywica akrylowa, właściwości mechaniczne.

Poly (methyl methacrylate) acrylic resin, first introduced as a denture base material in 1935, was successfully used in restorative dentistry over the past 70 years. However, the relatively low mechanical strength of this material and the resulting fracture of the denture remains an unresolved problem [1–3]. Over the years, several attempts have been made to improve the mechanical properties of acrylic resins, including the addition of various types of fibers: glass, polyethylene, polycarbonate, silica, ceramic and aramid [4–7]. These fibers can vary in diameter, length, form and arrangement in the fiber matrix bond. They can be used as reinforced materials with hot and cold curing methacrylic resins [8, 9].

Although glass fiber is transparent inside the polymer mass, the fiber bundles are very rigid and it is difficult to form them into different shapes. It is also necessary to silanize them before use and afterwards cover them with resins to protect from unraveling [3, 6]. The disadvantage of carbon fibers is their undesirable black color, which can change the color of the entire denture [1, 6, 9]. Polyethylene fibers are very interesting from a mechanical point of view, but they require the use of cold plasma arc for surface activation. After such a process, new groups can form on the surface of fibers and they can react with other resins, especially with the methacrylic groups [4, 5, 10]. Aramid fibers have been introduced for reinforcing the polymers because of their high mechanical properties, thermal resistance and limited solubility in different solutions [11, 12].

Various suggestions have been made to improve pretreated fiber surface and/or acrylic resin adhesion between fibers and denture base material: sandblasting, silanization, the application of different adhesive agents, plasma treatment and other pretreatment methods [13–17]. However, failure at the interface between acrylic resins and the fibers is still an unsatisfactorily resolved problem [3, 13, 18].

Unidirectional fibers used as reinforced materials consist of bundles of up to 12000 single fibers. Long fibers give anisotropic mechanical properties to acrylic resins, and are suitable for application in extremely high stress regions. Networks of fibers are used for removable whole or partial dentures, and unidirectional fibers are utilized for fixed partial dentures, splints and retainers [5, 19].

Fiber orientation has significant influence on the mechanical properties of the whole material such as its strength, elastic modulus and flexural deflection. Fibers parallel to the tensile force double the breaking resistance of the materials, but perpendicular fibers have no such influence. In that latter case the mechanical properties deteriorate [1, 3, 6, 7, 10, 14, 19]. Another factor affecting the strength of material is the quantity of fibers. An increase in the quantity of fibers into the polymer matrix enhances the flexural properties of the resins [3, 9]. The strength of the acrylic also depends on the place of the fibers inside the samples and the direction of the bending force. The quantity of absorbed water in a polymer matrix influences the flexural properties of the fiber reinforced polymers because of the plasticizing effect of water [20–22].

The very important factor is the adhesion between fiber and the polymer matrix [3]. With glass fiber, the solution is simplified by the silanization of the glass. But aramid fibers have the formula of polyparaphenylene terephthalamid. This material has aramid groups with strong hydrogen bonds instead of hydroxyl groups like glass or ceramic fibers. Therefore it is necessary to use another methacrylic monomer that can form strong hydrogen bonds with AF. When Kevlar is spun, the resulting fiber has great tensile strength (ca. 3 000 MPa), and a relative density of 1.44. When used as a woven material, it is suitable for mooring lines and other underwater application objects [20, 21].

The modified PMMA base resins system for glass fibers has been presented previously, but not with combination between aramid fibers with methacrylic monomers on their surface [18]. This system is clearly known in application with glass fibers, it consists of high weight molecular monomers and poly (methyl methacrylate). This mixture can remain in fluid state during storage and handling until polymerization with acrylic resin has been successfully finished [9, 10, 17, 23, 24].

The aim of the present study was to investigate the effect of water storage on the flexural properties of cold curing poly (methyl methacrylate) denture base resin reinforced with different surface pretreated aramid fibers placed in various forms and positions in the acrylic resin.

Material and Methods

Material

Test specimens were made of pouring methacrylic resins (Colacryl TS 2027®, Lucite) as two phases:

- liquid – 0.5% dimethyl-p-toluidyne (DMPT) and 99.5% methyl methacrylate (MMA) stabilized with 50 ppm hydroquinone (HQ),
- powder – contains poly (methyl methacrylate) polymer with citrate plasticizers, main particles granulation 40 μm , with 0.4% of benzoyl peroxide (BPO), and 0.5% 1-benzyl-5-phenyl barbituric acid on the surface.

Unidirectional monofilament aramid fibers Tawaron® (Teijin) which contain 99,9% poly para-phenylene terephthalamide were used for acrylic resin reinforced. According to the manufacturer, the physical and mechanical properties are: 18 μm diameter, bundles 2 mm bright, and 0,2 mm tick. The materials and chemicals used in this study are presented in Table 1.

Preparation of the Specimens for Study of Mechanical Properties

Preparation of Aramid Fibers

The poly (methyl methacrylate) solution to cover the surface of fibers were prepared by dissolving 1 g of methacrylic polymer Colacryl D 80FC (high

molecular weight) with 9 ml acetone. After mixing polymer with acetone, the solution was stirred for 3 hours at 23°C till complete disappearance of the polymer granulate. One meter of AF bands was covered with such solution by immersing them for 5 min and drying by air for 1 hour, for acetone evaporation.

Samples Preparation

Tests specimens with dimensions 3.3 ± 0.1 , 10.0 ± 0.1 and 65.0 ± 0.1 mm were prepared in stainless steel molds [18]. The surface of unidirectional AF covered by PMMA was slightly damped with methyl methacrylate and left under polyethylene foil for five minutes for effective monomer penetration. At the end, the fibers were placed inside metal molds. In the meantime, 14 g of Colacryl TS 2027 (self curing AR) was mixed with methacrylic monomer in separate silicon cup. After 30 seconds, each mold with aramid fibers was filled with the slurry of acrylic resins. The forms with acrylic resins were covered on both sides with two metal slabs.

Group 1 (from A to D) involves different fiber sizes and placements inside the samples:

- Group A. Two stripes of AF in lower part of acrylic sample, parallel to the longer axis of the specimens;
- Group B. Two stripes of AF in upper part of acrylic sample, parallel to the longer axis of the specimens;

Table 1. Materials and chemicals evaluated in the study

Tabela 1. Materiały i środki chemiczne oceniane w badaniu

Brand (Rodzaj)	Codes (Kod)	Batch no. (Numer serii)	Manufacturer (Wytwórca)
Methyl methacrylate – 99% (Metakrylan metylu – 99%)	MMA	33137272/1-16/12	Evonik-Degussa
Aramid fibers monofilament 5 mm width (Włókna aramidowe jednowłókowe o 5 mm szerokości)	Tawaron 1000	01-001 M nr 68.617	Teijin Corporation
Dimethyl-p-toluidine, 99% (Dimetylo-p-toluidyna, 99%)	DMPT	6860054	Sigma-Aldrich
Methacrylic oligomer with polycarboxylic acid group (Metakrylowy oligomer z grupami polikarboksylowymi)	Genomer 7154	320608178	Rahn
Urethane dimethacrylate, UDMA (Dimetakrylan uretanu, UDMA)	Plex 6661-0	1260991096	Evonik-Degussa
Bisphenol A glycol dimethacrylate, Bis GMA (Bisfenol A glikol dimetecrylat, Bis-GMA)	Plex 6662	3447152	Evonik-Degussa
Colacryl D 80 FC with 0.28% benzoyl peroxide (Calocryl D 80 FC z 0,28% nadtlaniem benzoilu)	PMMA	1429308	Lucite
Colacryl TS 2027 with 0.4% benzoyl peroxide and 0.5% 1-benzyl-5 phenyl barbituric acid (Colacryl TS 2027 z 0,4% nadtlaniem benzoilu i 0,5% kwasem 1-benzyl-5 fenylobarbiturowym)	PMMA	B3/17247	Lucite
Acetone, 99% (Aceton, 99%)		57100206	Chempur

- Group C. Two 6 cm long aramid stripes were cut in to small pieces 5 mm in length with scissors and mixing with acrylic powder before mixing with monomer;

- Group D. Six 9 mm long stripes were placed on lower part of the sample perpendicular to the long side.

In Group 2 (from E to G), samples were prepared with different kinds of methacrylic high molecular weight monomers dissolving 3 g of resin in 3 g of acetone to decrease their viscosity. In all groups of samples, two fibers strips were put on the lower part of the samples width:

- Group E. Genomer 7154 is methacrylic oligomer with polycarboxylic acid group. According to the manufacturer, the product is used for adhesive painting on glass and metal surfaces. Final concentration of the monomer was 10% of the fiber mass.

- Group F. Plex 6661-0 (UDMA) is aliphatic urethane dimethacrylate. It was used at 10% concentration of the fiber mass;

- Group G. Plex 6662 (bis GMA resin) has aromatic structure with two methacrylic groups. It was used at 10% of the fiber mass. The concentration of the remaining resins on the fiber surface was checked with laboratory balance, after 24 hour, when acetone evaporated.

Samples in Group 3 (from H to J) were prepared with completely dissolving 1 g PMMA polymer (D 80FC) in 10 g acetone. One gram of each methacrylic monomers used in previous experiments was added to each sample. The final concentration of polymethyl methacrylaceton and methacrylic monomers on the surface of fibers equalled 5%. It was checked at laboratory balance after 24 hours when acetone had completely evaporated.

- Group H. Mixture 1 : 1 PMMA (D 80FC) with Genomer 7154;

- Group I. Mixture 1 : 1 PMMA (D 80FC) with Plex 6661-0;

- Group J. Mixture 1 : 1 PMMA(D 80FC) with Plex 6662-0.

Each of the molds filled with methacrylic resins and aramid fiber and covered with metal plate, was placed in pressure kettles (Zhermapol, Poland) with water temperature of 65°C, pressure 2.5 bar and kept at this temperature for 30 minutes. After complete curing, the samples were removed from the molds and ground wet with grinding paper P1200 (15,3 µm, silicon carbide paper Norton Vulcan, Saint Gobain, Kolo, Poland) [17].

For each group (from A to J), 12 samples were prepared and divided into two groups (120 samples for the whole experiment). Half of the samples (6 samples) of each group were stored in to the

water-bath at 37° ± 1°C for 7 days, and the second one was kept at air 23° ± 1°C and humidity 50% for 24 hours.

Flexural Properties of Methacrylic Polymer with Aramid Fibers

Flexural properties of dry and wet specimens were determined using a three – point bending test with Instron 4411 testing machine H 2097 (Instron Corp., England) and Series IX Automated Materials Testing System Version 5.34.00. computer program. The three-point bending test was carried out in water immersion at 37° ± 1°C. The specimens were bent in a three-point transverse testing rig with 50 mm between the two supports and with a constant cross head speed of 5.0 mm/min [18]. The ultimate transverse strength (δ) was calculated from the equation:

$$\delta = -\frac{3FL}{2bh^2},$$

where 'F' is the applied load (N) at the highest point of the load – deflection curve, 'L' is the span length (50.0 mm), 'b' is the measured width of the test specimen, and 'h' is the measured thickness of the test specimen. The dimensional measurements were made with digital micrometer Links (Japan), (standard measurement uncertainly 0.01 mm).

The flexural modulus (E) was calculated from the equation:

$$E = \frac{l^3F}{4bh^3d},$$

where 'd' is the deflection corresponding to load 'F' at the point in the straight line portion of the trace.

Statistical Analysis

The results of the measurements of ten different groups were investigated by independent sample T test at 95% significance level using a statistical software package (SPSS, Statistical Package for the Social Sciences for Windows, 15.0.1, Chicago, IL, USA).

Results

Ultimate flexural strengths (δ) and flexural module (E) of aramid fibers polymers before and after water immersion for different positions of the

fibers inside the samples are presented in Table 2. The same values for modification of the samples with different acrylic monomers can be found in Table 3. The flexural properties of acrylic resins generally improve with the use of aramid fibers but it depends on the position of the fiber inside the sample. An increase in the flexural strength is possible if the fibers are in opposite direction to the breaking force.

Best results were obtained with Bis GMA as an addition resins to PMMA. Another interesting composition contains Genomer 7154. Immersion in water for 7 days slightly decreases the mechanical properties of unfilled acrylic resins, but for specimens with aramid fiber it has a smaller effect. There was no statistically significant difference in the measured intimate flexural strength and flexural modulus between unfilled acrylic resins and these with 0.5 cm length aramid fiber mixed with of the samples.

Discussion

This study was designed to evaluate the effect of different orientation and various kinds of acrylic high molecular weight monomers on the flex-

ural properties of aramid fibers reinforced acrylic resins (AFRAR) [25–27]. The results of the present investigation indicate the importance of position of AF inside the sample. If aramid bands are cutting into short segments they haven't big effect on improving the mechanical properties, however flexural resistance depends on the environmental conditions.

Uzun and Keyf used 5 mm long various fibers inside the samples and obtained good results for all of them. AF have a mean flexural strength about 849.0 MPa, where a control group acrylic resins samples without reinforcement 696.0 MPa [28]. The dimensions of the samples were the same as ISO Standard, but the cross head speed during the breaking process was smaller 2 mm/min instead of 5 mm/min [22]. All the specimens exhibited lower transverse strength with an increase water immersion time.

Fiber position inside the sample is very important. It is possible to increase the flexural strength if the fibers are in opposite direction to the breaking force. They can bend and absorb the energy without destruction of the resins. It is possible to obtain an increase in the breaking resistance of about 100% compared to samples without rein-

Table 2. Flexural strength aramid fiber-reinforced acrylic resin depending on different fiber position inside the sample

Tabela 2. Wytrzymałość na zginanie tworzywa akrylowego wzmocnionego włóknem aramidowym umieszczonym w różnych miejscach w próbce

Sample (Próbka)	*FS dry (*FS suche) MPa	FS wet (FS mokre) MPa	**MD dry (**MD suche) mm	MD wet (MD mokre) mm	***Y dry (***Y suche) MPa	Y wet (Y mokre) MPa
Colacryl TS 2027 (Colacryl TS 2027)	80.4 (4.2)	74.2 (3.6)	4.43 (0.63)	4.98 (0.49)	2417.1 (6.5)	2287.3 (2.4)
Colacryl TS 2027 + 2 AF in lower part of sample (Colacryl TS 2027 + 2 włókna aramidowe w dolnej części próbki)	134.2 (5.5)	127.2 (5.1)	6.34 (0.45)	6.67 (0.38)	6234.2 (3.5)	6005.3 (3.2)
Colacryl TS 2027 + 2 AF in upper part of sample (Colacryl TS 2027 + 2 włókna aramidowe w górnej części próbki)	82.1 (8.5)	74.8 (7.6)	5.02 (0.49)	5.76 (0.12)	3100.0 (2.4)	2987.3 (2.1)
Colacryl TS 2027 + 1% AF 5 mm long in all direction (Colacryl TS 2027 + 1% włókien aramidowych o długości 5 mm w różnych kierunkach)	86.2 (4.2)	80.1 (5.9)	4.98 (0.55)	5.33 (0.23)	2658.3 (3.6)	2340.2 (3.7)
Colacryl TS 2027 + AF perpendicular to the long side (Colacryl TS 2027 + włókno aramidowe ułożone pionowo)	76.0 (8.2)	77.3 (7.1)	4.23 (0.22)	4.89 (0.32)	2560.4 (4.6)	2404.1 (3.5)

*FS – flexural strength.

**MD – maximal displacement.

***Y – Young module.

*FS – wytrzymałość na zginanie.

**MD – maksymalne odgięcie.

***Y – moduł Younga.

Table 3. Flexural test of aramid fiber-reinforced acrylic resin with fibers covered different kind of methacrylic resins**Tabela 3.** Testy wytrzymałości próbek akrylowych wzmocnionych włóknem aramidowym pokrytym różnymi rodzajami żywic metakrylowych

Sample (Próbka)	*FS dry (*FS suche) MPa	FS wet (FS mokre) MPa	**MD dry (**MD suche) mm	MD wet (MD mokre) mm	***Y dry (***Y suche) MPa	Y wet (Y mokre) MPa
PMMA (Polimetakrylan metylu)	134.2 (5.5)	127.2 (5.1)	6.34 (0.45)	6.67 (0.38)	6234.2 (3.5)	6005.3 (3.2)
Genomer 7154	154.2 (6.1)	144.1 (4.5)	6.71 (0.27)	7.12 (0.37)	7905.1 (23.2)	7708.3 (5.7)
Plex 6661-0	140.1 (5.2)	133.3 (5.9)	7.41 (0.43)	7.80 (0.41)	6560.9 (9.4)	6300.2 (7.1)
Plex 6662	163.5 (7.5)	155.2 (2.9)	7.06 (0.25)	7.38 (0.36)	8700.2 (8.5)	8540.8 (6.3)
PMMA 5% + 5% Genomer 7154	149.1 (7.3)	144.2 (7.3)	6.58 (0.23)	6.89 (0.49)	7800.0 (5.5)	7456.0 (4.2)
PMMA 5% + 5% Plex 6662-0	158.2 (3.5)	150.8 (3.1)	7.72 (0.54)	8.12 (0.14)	78045.0 (3.5)	7530.1 (3.7)
PMMA 5% + 5% Plex 6661-0	136.1 (4.6)	127.2 (3.8)	5.93 (0.23)	6.32 (0.23)	6004.1 (2.4)	5897.2 (4.1)

*FS – flexural strength.

**MD – maximal displacement.

***Y – Young module.

*FS – wytrzymałość na zginanie.

**MD – maksymalne odgięcie.

***Y – moduł Younga.

forcement [29–31]. But if the fibers are positioned in the upper part of the sample close to the breaking head, there is no improvement in flexural parameters.

The efficiencies of fiber reinforcement in different orientations are described for the direction of tensile loading [4, 6, 7, 32, 33]. However, in flexural loading, tensile, compression and shear loadings are all combined, with compression in the upper part, and with tension in the lower part of the samples. Middle parts of the specimens are subject to the influence of shear forces [18, 19]. These results are similar to those of other researchers. Göhring et al. [14] observed a significant reinforcing effect of glass fibers when they were placed on the tension side, but not when placed on the compression side.

An improved effect for aramid fibers was observed by Uzun et al. [28, 29] when the fibers were in the middle layer of the samples. The maximum strength was 442 MPa for reinforcement specimens and 400 MPa for unfilled acrylic resins. These results were obtained for the same cross head speed during break, but the samples have thickness of 4 mm instead of 3.3 according ISO standards [18]. If the fibers are placed in perpendicular to long side of the sample, they have a destructive effect on the mechanical properties. It is clearly visible in the group immersed in the water for 7 days. It could be connected with the penetration of water along the fibers. The specimens were broken in the part with perpendicular bundles of fibers.

Previous studies with bidirectional glass fibers showed the reduction in ultimate flexural

strength after long water sorption [21, 22, 28, 32–34] approximately half of the initial value for glass fibers sized with cross linked PMMA and composite materials. But glass fiber contains OH groups not covered with silan and this can make them more susceptible to water (silan hydrolyzation process in water).

Adding other acrylic monomers has an effect on the mechanical properties of the samples. The best results are obtained with Bis GMA (Plex 6662) monomer as addition to PMMA resins. This resin has two OH groups and can form strong hydrogen bonds with AF. The structure of bis phenol backbone can make this resin very resistant and a good cross linker. It is commonly used in dental composite. Another interesting composition contains Genomer 7154. This resin with acid groups can make strong hydrogen bonds with aramid. It has a long aliphatic chain between reactive methacrylic groups which can make the material more flexible. Nonetheless increased flexural strength is clearly visible in this group compared to unfilled resin. Plex 6661-0 is an aliphatic dimethacrylate which can form weaker hydrogen bond with AF compare to acid groups in Genomer, and makes the entire structure more flexible, hence important for removable dentures.

Standard deviation in flexural properties within groups may partly be attributed to specimen variation. Even if the same laboratory process is followed in preparation of specimens, there are several undesirable factors causing specimen variations during preparation, such as wetting aramid surface by the polymer and monomers, PMMA

penetration between the fibers, microporosity on the border of fiber and polymer or sizing process. However, differences between specimens can best be explained by placement of fibers.

The authors concluded that unidirectional

aramid fibers with their surface pretreated with methacrylic polyfunctional monomers, may find application in restorative dentistry due to their contribution to increased mechanical properties of acrylic resins as denture base materials.

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