POLIMERY

The influence of quaternary ammonium salts on mechanical properties of light-cured resin dental composites

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Abstract: The composite consisting of bis-GMA/UDMA/HEMA/TEGDMA monomer mixture forming a polymer matrix filled with silanized silica (45 wt%) was modified with CTAB or DODAB quaternary ammonium salts (0.5–2.0 wt%). The hardness, flexural strength as well as diametrical tensile strength of the composite before and after modification were evaluated. The type and amount of salt affected the hardness, flexural strength, and shrinkage stress; however, they did not influence the diametral tensile strength of the tested composites.

Keywords: dental composites, quaternary ammonium salts, mechanical properties

Wpływ czwartorzędowych soli amoniowych na właściwości mechaniczne fotoutwardzalnych kompozytów dentystycznych

Streszczenie: Kompozyt składający się z mieszaniny monomerów bis-GMA/UDMA/HEMA/TEGD-MA tworzących osnowę polimerową napełnioną silanizowaną krzemionką (45% mas.) modyfikowano czwartorzędowymi solami amoniowymi CTAB lub DODAB (w ilości 0,5–2,0% mas.). Zbadano twardość, wytrzymałość na zginanie oraz wytrzymałość na średnicowe rozciąganie kompozytu przed i po modyfikacji. Rodzaj i ilość soli miały wpływ na twardość, wytrzymałość na zginanie oraz naprężenia skurczowe, nie wpłynęły natomiast na wytrzymałość na średnicowe rozciąganie badanych kompozytów.

Słowa kluczowe: kompozyty dentystyczne, czwartorzędowe sole amoniowe, właściwości mechaniczne.

Currently, the most used materials in dentistry (reconstructive and prosthodontics) are resin composite materials. With their use, one can recreate missing teeth and restore the proper functioning of the speech apparatus. Importantly, these materials ensure the appropriate mechanical properties while meeting aesthetic requirements as well as maintaining biocompatibility [1]. Composite materials can be used, except for direct reconstruction, to make several types of prosthetic restorations, such as inlays, onlays, overlays, crowns, and veneering elements [2, 3]. Resin dental composites (RDC), despite their advantages, exhibit drawbacks, such as polymerization shrinkage, contraction stress and affinity to surface plaque accumulation. This unfortunately creates ideal conditions for the occurrence of bacterial and fungal invasions [4]. Overall, this phenomenon can lead to mechanical and chemical degradation and promote the appearance of secondary caries. Secondary caries is the

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most common reason for restoration replacement, which leads to a reduction in dental tissue as well as unnecessary costs [5]. Hence there is a need for modifications of composites with biostatic and/or biocidal substances.

An example of a potentially biostatic and/or biocidal modifiers are quaternary ammonium compounds (QACs). These are substances commonly used as disinfectants. They are popular due to their good thermal stability as well as lack of odor and color. Depending on their concentration, they show activity against bacteria, spores, mycobacteria, algae, fungi, and viruses, among others [6–8]. The antibacterial action of quaternary ammonium salts is based on the disruption of the electrical balance of the bacterial cell membrane. Positively charged QACs in contact with the negatively charged cell membrane cause a break in its continuity, leading to the release of K⁺ ions and other cytoplasmic components. The result of this phenomenon is the death of the microorganism [9–11].

Material modifications with different quaternary ammonium compounds are also visible in dentistry biomaterials. Modification of polymethylmethacrylate (PMMA), a material used in the manufacture of dentures, with QAC, was proposed by Lee et al. [12]. This modification enables PMMA to acquire biocidal properties against Escherichia coli as well as Streptococcus mutans and has no negative effect on flexural strength. Songa et al. [13] also modified PMMA base materials with the quaternary ammonium salt of chitosan and showed that there was no notable change in tensile strength and cytotoxicity with maintained antibacterial properties. Cheng et al. [14] incorporated a new quaternary ammonium monomer (dimethylaminododecyl methacrylate, DMADDM) into a dental primer and an adhesive to study the effects on antibacterial properties and bonding to dentin. It was shown that the addition did not adversely affect dentin bond strength. In commercially available dental adhesive systems, one can nowadays find quaternary ammonium units, i.e., 12-methacryloyloxy dodecypyridinium bromide (MDPB) in Clearfil Protect BondTM (Kuraray Co. Ltd., Japan) [15]. Also, the low-viscosity ionic dimethacrylate (IDMA) monomers containing quaternary ammonium groups prone to copolymerization with other monomers exhibit antibacterial properties and are proposed for dental use [16]. Research was also conducted on the use of an experimental composite resin containing a urethane dimethacrylate quaternary ammonium compound (UDMQA-12). The study showed that the experimental composite was biocompatible, while its mechanical properties were like some commercially available resin composites. It may be useful in preventing the formation of secondary caries [17]. Fugolin et al. [17] synthesized tertiary quaternary ammonium acrylamides (AM) with methacrylamides (MAM) and incorporated them into an experimental composite. Research showed that these materials exhibited strong antibacterial properties, and their mechanical properties were like those of the control group [18]. Dekel-Steinkeller et al. [19] investigated the properties of a new bulk-fill flowable composite (InfinixTM, Nobio Ltd., Israel) that contains quaternary ammonium silica (QASi) filler particles. They demonstrated the antimicrobial activity of QASi is comparable to the properties of quaternary ammonium polyethyleneimine (QPEI) particles [19]. The biocidal activity of dimethylaminoethyl methacrylate quaternary monomer (DMAEMA-BC) and hydrophobic POSS nanoparticles (Triazolium-POSS) with built-in bactericidal functional groups such as 1,2,3-triazolium were also investigated. Composite with Triazolium-POSS showed higher bactericidal activity against Streptococcus mutans [20]. Munguía-Moreno et al. [21] modified two resin-modified glass ionomer cements (RMGICs) with benzalkonium chloride, cetylpyridinium chloride, hexadecyltri-methylammonium bromide or silver nanoparticles and determined the cytotoxicity, surface roughness, microhardness of materials. Modification of GC Fuji ORTHO LC and GC Fuji ORTHO PLUS glass ionomer cements with CTAB influenced hardness and roughness of materials depending on the amount of salt and the material to be modified [21].

CTAB exhibits particularly good antibacterial and antistatic properties. It shows biocidal activity against some Gram-negative bacteria and against Gram-positive bacteria. DODAB salt influences the viability of *Candida albicans*. The use of micromolar concentrations of salt allows the destruction of bacteria, however in the case of candida, much higher concentrations of salt is required [22].

The purpose of this study was to investigate the influence of type and amount of quaternary ammonium salts (hexadecyltrimethylammonium bromide (CTAB), Fig. 1



Fig. 2. Dimethyldioctadecylammonium bromide (DODAB)



Fig. 3. Monomers used for matrix preparation

or dimethyldioctadecylammonium bromide (DODAB), Fig. 2) on the selected mechanical properties of experimental light-cured dental composites. The presented studies are preliminary.

EXPERIMENTAL PART

Materials

Four monomers – bisphenol A-glycerolate dimethacrylate (bis-GMA, purity \geq 97 %), urethane dimethacrylate (UDMA, purity \geq 97 %), 2-hydroxyethyl methacrylate (HEMA, purity \geq 97 %), triethylene glycol dimethacrylate (TEGDMA, purity \geq 95 %) (all from Sigma-Aldrich, St. Louis, USA) were used to make the matrix in the ratio of 40/40/10/10 wt% (Fig. 3).

As photo-initiator system, camphorquinone (CQ, purity \geq 97 %) in the amount of 0.4 wt%, co-initiator 2-(dimethylamine)ethyl methacrylate (DMAEMA, purity \geq 98 %) at 0.9 wt% and a stabilizer of the polymerization

process butylated hydroxytoluene (BHT, purity ≥99 %) at 0.1 wt% were used (Fig. 4). Weight percentages of photoinitiator system were calculated based on the weight of the monomer mixture. All ingradients of photo-initiating system are from Sigma-Aldrich, St. Louis, USA.

Then the matrix was filled with silica Arsil (Zakłady Chemiczne Rudniki S.A., Rudniki, Poland) silanized by 3-methacryloxypropyltri-methoxysilane (γ -MPTS, Unisil Sp. z o.o., Tarnow, Poland) in the amount of 45 wt%. To modify the composite CTAB or DODAB (both from Sigma-Aldrich, St. Louis, USA, purity \geq 98 %), in amounts of 0.5, 1.0 and 2.0 wt% (on composite weight), were incorporated. The samples were then made using silicone molds. After placing the material in the molds, a tape (Hawe Striproll, Kerr, Bioggo, Switzerland) was applied to the surface of the molds to prevent the formation of an inhibition layer. The samples prepared in this way were covered with laboratory slides and then cured for 20 s using THE CURE TC-01 polymerization lamp (SPRING; Norristown, Pennsylvania, USA) with power 1200 mW/cm² at 1.5 mm thickness of the material (Fig. 5, 6).



Fig. 4. Additional substances used to manufacture the experimental composite material

Methods

The effect of modifying the composite material with CTAB or DODAB salts on the Vickers hardness, flexural strength and diametrical tensile strength was investigated. The values of shrinkage stress generated during the photopolymerization of the composite were also determined (Tab. 1). Vickers hardness (HV) was determined using a ZHµ-2 semi-automatic hardness tester (Zwick/Roell, Germany), with a load of 10 N. Nine specimens were made for each test group, in the form of a cylinder with a height of 3 mm and a diameter of 6 mm. The flexural strength (FS) of the composite was determined on a three-point bending test (TPB). During this test also the flexural modulus (E_f) was assessed. It was conducted according to ISO 4049 [23] with six specimens for composite with dimensions of $25 \times 2 \times 2$ mm. The traverse speed during the test performed was 1 mm/min [24]. For diametral tensile strength (DTS) testing, the nine specimens (the same shape and dimension as for HV) are subjected to compression along the diameter, perpendicular to the major axis, until failure. During the test, the crosshead moved at a speed of 2 mm/min. An elasticoptical method using a Gunt FL200 circular polariscope (Gunt Gerätebau GmbH, Barsbüttel, Germany) was used to evaluate the shrinkage stress formed during photopo-



Fig. 5. Silicone mold filled with composite

lymerization of the composite material. Stress was calculated by using transformed Timoshenko formulas and elastic theory formulas – a more detailed description of the method was presented in our earlier work [25, 26].

The computer program Statistica v.13 (Tibco Software Inc., Krakow, Poland) was used for the statistical analysis. For the obtained test results, the arithmetic mean, standard deviation, median, maximum, and minimum were calculated. Based on the data obtained, graphs of the mean and median were made, depending on the study. Based on the Shapiro-Wilk test, the conformity of the distribution to the normal distribution was verified. In the absence of conformity to the normal distribution, the Kruskal-Wallis test (for independent samples) was used to confirm the hypotheses. Next, it was verified which groups were statistically different from the others using multiple comparisons of the mean ranks of all groups, in agreement with the assumption of an alpha level of significance of 0.05.

RESULTS AND DISCUSION

The obtained composite materials with different concentrations of antimicrobial modifiers, were tested for mechanical properties, as well as stresses during curing. The results are summarized in Table 2.



Fig. 6. Sample during curing

T a b l e 1. Characteristics of mechanical tests



T a b l e 2. Hardness (HV), diametral tensile strength (DTS), flexural modulus (E_{f}), flexural strength (FS) of bis-GMA/UDMA/ HEMA/TEGDMA (40/40/10/10) composites modified with CTAB or DODAB

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QACs amount wt%	HV	DTS MPa	FS MPa	E _f MPa	
0	33.0 ± 2.4	34.2 ± 2.4	92.4 ± 7.7	4108 ± 194	
CTAB					
0.5	25.8 ± 0.8	33.6 ± 10.8	75.3 ± 12.5	3514 ± 676	
1.0	25.0 ± 0.7	30.9 ± 7.6	77.4 ± 6.6	3078 ± 562	
2.0	24.7 ± 1.1	34.2 ± 3.0	74.7 ± 10.3	2716 ± 200	
DODAB					
0.5	25.7 ± 0.5	33.9 ± 5.3	78.6 ± 10.0	2862 ± 269	
1.0	24.8 ± 0.8	33.6 ± 4.6	73.7 ± 6.5	3050 ± 359	
2.0	29.4 ± 1.3	31.7 ± 5.4	68.7 ± 7.5	3664 ± 244	



Fig. 7. Hardness of the composites differing with CTAB content



Fig. 9. The effect of CTAB content on diametral tensile strength of the composites



Fig. 11. The effect of CTAB content on flexural strength of the composites



Fig. 8. Hardness of the composites differing with DODAB content





FS, MPa

80

75

70

65

Fig. 10. The effect of DODAB content on diametral tensile strength of the composites



Fig. 12. The effect of DODAB content on flexural strength of the composites



Fig. 13. The effect of CTAB content on flexural modulus of the composites

The highest hardness (HV = 29.4 ± 1.3) after modification with QACs was obtained for composite with 2 wt% of DODAB, while the lowest (HV = 24.8 ± 0.8) was observed with a concentration of 1 wt%. Modification with CTAB salt with a concentration of 0.5 wt% resulted in the highest values of hardness (HV = 25.8 ± 0.8), and the lowest value was obtained when the concentration was 2 wt% (Tab. 2). The Shapiro-Wilk test showed inconsistency with the normal distribution for two DODAB measurements and two CTAB measurements (p<0.05). The Scheffe test showed significant statistical differences between the hardness results of the unmodified material and the material modified with 0.5 wt% CTAB, as well as the unmodified material and the composite subjected to 1 wt% CTAB modification. Analysis of the DODAB-enriched composite data showed statistically significant differences between the unmodified composite and the composite with 0.5 wt% DODAB. Differences were also observed between the values obtained when evaluating the hardness of the unmodified composite and the composite with 1 wt% DODAB. Statistically significant differences were also shown for the composite with 1 and 2 wt% DOAB (Figs. 7, 8).

The highest values of diametral tensile strength were obtained with 2 wt% of CTAB salt and 0.5 wt% of DODAB salt. Modification of 1 wt% with CTAB salt gave the lowest diametral tensile strength values, while in the case of DODAB salt, the lowest values were achieved with 2 wt% salt. The Shapiro-Wilk test showed the presence of inconsistency with the normal distribution. This was the case for measurements of the CTAB-modified material and two DODAB-modified (p<0.05). No statistical differences were observed for the DTS of the CTAB salt-modified composite material. The Kruskal-Wallis tests also showed no statistically significant differences between the DTS results of the unmodified and DODABmodified composite material (Fig. 9, 10).

The composite without QACs had the highest FS (92.4 MPa) and Ef (4108.0 MPa). After modification the



Fig. 14. The effect of DODAB content on flexural modulus of the composites

lowest values of flexural strength were achieved for the composite with 2 wt% of CTAB (FS = 74.7 ± 10.3 MPa), while the highest values were obtained in the case of the concentration of 1 wt% (FS = 77.4 ± 6.6 MPa) (Tab. 2). For composites with DODAB the highest strength characterized composite with amount 0.5 wt% (FS = 78.6 ± 10.0 MPa), the lowest gained composite with 2 wt% (FS = 68.7 ± 7.5 MPa). Modification of the composite material with the quaternary ammonium salt DODAB, with increasing its concentration, causes a decrease in flexural strength (Tab. 2). The Shapiro-Wilk test showed inconsistency with the normal distribution for measurements of the CTAB-modified material, as well as the DODAB (p<0.05). The Tukey test showed statistically significant differences between FS results for the unmodified and modified with 2 wt% CTAB composites. Scheffe's test showed statistically significant differences in FS between the composite without CTAB and with 2 wt%. Statistically significant differences were also shown for the FS of the unmodified material and the composite with 1 wt% of DODAB, as well as between the unmodified composite and the composite with 2 wt% of DODAB (Fig. 11, 12).

The flexural modulus of the composite material decreases with increasing CTAB content (Fig. 13). The opposite behavior was observed for DODAB (Fig. 14). The Shapiro-Wilk test showed inconsistency with the normal distribution for the CTAB and the two DODAB measurements (p<0.05). Scheffe test showed statistically significant differences between the values of the elastic modulus of the unmodified composite and the composite with 1 wt% of CTAB and the unmodified composite and the composite with 2 wt% of CTAB. The Kruskal-Wallis test showed significant statistical differences between the elastic modulus values for the unmodified material and the material enriched with the addition of 0.5 wt% DODAB. In addition, there were also significant statistical differences for the flexural modulus values of the unmodified material and the composite with 1 wt% of DODAB.

QACs amount wt%	σ _r MPa	σ _Θ MPa	σ _{int} MPa			
0	5.6 ± 0.4	-6.9 ± 0.5	12.6 ± 0.8			
CTAB						
0.5	6.0 ± 1.2	-7.3 ± 0.9	13.3 ± 2.1			
1.0	5.9 ± 1.0	-7.4 ± 1.1	13.2 ± 2.2			
2.0	7.7 ± 0.0	-8.7 ± 0.1	16.3 ± 0.1			
DODAB						
0.5	6.1 ± 0.3	-7.2 ± 0.4	13.3 ± 0.7			
1.0	7.3 ± 0.4	-8.6 ± 0.5	15.9 ± 1.0			
2.0	5.7 ± 0.5	-6.9 ± 0.6	12.6 ± 1.0			

T a ble 3. Effect of CTAB and DODAB salts on shrinkage stress formed during polymerization

 σ_r – radial stresses [MPa]; σ_{Θ} – circumferential stresses [MPa]; σ_{int} – reduced stresses [MPa]

The lowest values of shrinkage stresses were recorded when the antimicrobial additive DODAB with a concentration of 2 wt% and CTAB with a concentration of 1 wt% was added to photopolymerized composites. Composite with 1 wt% of DODAB or with 2 wt% of CTAB generated the highest contraction stress during polymerization (Tab. 3, Fig. 15).

The aim of the research was to develop an innovative material with biocidal and/or biostatic properties (due to the presence of CTAB or DODAB) and good mechanical performance. The hardness measurement of the experimental composite showed that the modification with CTAB or DODAB leads to a reduction of this parameter when compared with the control. The highest hardness composite with CTAB amounted to approx. 26 and for the composite with DODAB approx. 29. Similar hardness values of experimental dental resin composites can be also found in other studies [27–29]. Based on the literature, the minimum Vickers hardness value of commercial composite materials

was found to be between 40 and 50 [28, 30, 31]. The value of the hardness parameter may also be an effect of a low filler concentration in our experimental materials. It should be remembered that the hardness rises with the filler content of the composite [29, 32-34]. Worth pointing out is HV of material modified with 2 wt% of DODAB (29.4 ±1.3), however, the reason for it could be the uneven distribution of the antibacterial modifier in the material. The decrease in mechanical properties observed in our study may relate to the increasing ratio of organic to inorganic phase and decreasing in the total amount of monomers prone to photopolymerization (CTAB nor DODAB does not participate in network formation). Worth pointing out is also the total amount of CTAB or DODAB incorporated into the experimental material which is up to 2 wt% per whole mass of the composite. CTAB or DODAB (formula weight 364.45 g/mol and 630.95 g/mol, respectively) also could act as spatial hindrance between monomers resulting in lower conversion degree and worsened mechanical properties.



Fig. 15. Images of isochromes visible in epoxy plate after polymerization: a) - before modification, b) 0.5 wt% CTAB, c) 0.5 wt% DO-DAB, d) 1 wt% DODAB, e) 1 wt% CTAB, f) 2 wt% CTAB, g) 2 wt% DODAB

Due to chemical structure and hydrophilic character, quaternary ammonium salts (especially quaternary ammonium methacrylate) can act as plasticizers [35, 36] due to their chain structure. QAM's chains can easily attach to them resulting in polymer chains separation and larger spaces between them, which overall can lead to movement relative to each other. The effect of this process could increase the ductility of the material.

The obtained composite, due to its low hardness, might find its potential used as a lining material, or for the creation of restorations of deciduous teeth, or as class V fillings [37, 38].

The minimum value of diametral tensile strength for dental composites should not be less than 24 MPa [39, 40]. The composite with or without CTAB or DODAB met the above standard. The mechanical strength is one of the most important aspects that is considered when selecting the type of material for tissue replacement or prosthetic restoration. Durable materials are more resistant to fracture and deformation, which has a significant impact on achieving clinical success [41, 42].

Testing of the three-point bending-based flexural strength showed a downward trend in the material's toughness. The highest FS values were shown for both composites with 0.5 wt% concentration of DODAB or CTAB. Consecutively, strength values of approx. 79 MPa were obtained for DODAB modified composite, and approx. 77 MPa for composite with CTAB. The reduced strength, apart from material aspects mentioned above, may be due to the shape and dimension of the samples used for the test. The large size of the specimens may cause uneven polymerization and crosslinking, which eventually may lead to the generation of shrinkage stresses under exposure. Nevertheless, it should be concluded that flexural strength of composites with DODAB or CTAB do not deviate from flexural strength of some commercial composites [43, 44]. They also are within the accepted criteria for polymeric dental composites, where the material must achieve a strength of 80 MPa for loaded (chewing) surfaces and 50 MPa for less loaded areas [23]. Maintaining the proper level of strength of the composite material is an important aspect. In the case of the tested experimental composite, slight deteriorations in mechanical properties are visible, but those are not high disturbances as the lack of statistical differences is observed, for example between the unmodified material and the modified composite with DODAB or CTAB, with a concentration of 0.5 wt%. Depending on the obtained strength value of the material, a selection is made for the appropriate restoration. Notably, high strength is important for filling Class I, II, and IV cavities. Inadequate strength can generate the appearance of clusters of marginal and volumetric cracks [37, 45].

The results of elastic modulus in bending showed that the enrichment of the material with the DODAB or CTAB made composites less stiff. Lower elastic modulus after modification is a consequence of disruption the organic/inorganic phase ratio (with a predominance of the organic phase) [46]. On the other hand, composite with 2 wt% concentration of DODAB resulted in the highest value of the parameter from all modified composites. This is a positive development, since increasing the elastic modulus will reduce the formation of potential deformation of the material [47]. The different effects of the type of salt on the elastic modulus and the observed trends, increase in E_f with DODAB increase and the other way round when CTAB is used, may be due to the difference in structures of the CTAB and DODAB molecule. The CTAB molecule has a shorter, single carbon chain, whereas DODAB molecule has longer double chain.

When polymeric materials are cured, shrinkage stresses are generated due to polymerization shrinkage of the composite. Through shrinkage of the material, microleakage can occur at the boundary between the restorative material and the tooth. The consequence of this phenomenon can also be hypersensitivity as well as discoloration on the restored surface. The formation of such a gap is an ideal place for the growth of bacterial colonies and the formation of biofilm, which eventually can lead to the development of secondary caries, as well as mechanical and chemical degradation of the material. It may become necessary to replace the restoration, which generates additional costs, and, more importantly, possibility of tissue loss [48].

Analyzing the data obtained during the study of shrinkage stresses showed that their values ranged from 12.6 to 16.3 MPa. These results are within the range of stresses generated by commercial composite materials, which vary between 0.3 and 24 MPa [48–51]. It can be concluded that both DODAB and CTAB salt modifications affect the generation of shrinkage stresses. In the case of DODAB, the highest values are obtained at a modifier concentration of 1 wt% and 2 wt% for CTAB salt. In the case of modification with CTAB, it can be observed that as the modifier concentration increases, the shrinkage stress in the material also increases. Note that a low filler concentration was used for this experimental composite. The enhancement of the ratio of organic to inorganic phase may have been the reason for the increase in shrinkage stress.

CONCLUSIONS

Bis-GMA/UDMA/HEMA/TEGDMA monomer-based composites with silanized silica and CTAB or DODAB quaternary ammonium salts were synthesized. The addition of salts reduced the hardness, but did not affect the diametral tensile strength, the value of which met the minimum requirements for this type of composite materials. The flexural strength decreased with increasing salts content. The addition of salts affected the shrinkage stresses generated during polymerization. The lowest shrinkage stress was recorded at 2 wt% DODAB and 1 wt% CTAB content. Composite with 1 wt% of DODAB or with 2 wt% of CTAB generated the highest shrinkage stress. Further research should be conducted using a composite with an increased amount of filler or a different type of filler. The next article in preparation will describe the biocidal/biostatic properties of the obtained composites. Equally important is the study of the cytotoxicity of antibacterial additives, due to the considerable amount of literature reports on the potential harm of QAC salts to the human body [52–54]. The effect of antibacterial modification on the photopolymerization process and degree of crosslinking of the composite should be investigated, and aging studies should be conducted as well.

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