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The influence of filler amount on selected properties of new experimental resin dental composite

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Abstract: Aim of the study was to evaluate the influence of filler amounts on mechanical properties and contraction stress of light-curing experimental composite. Hardness, flexural strength, diametral tensile strength of material filled with 40, 50 or 60 wt. % of silanized silica were tested. The contraction stress was measured after 24 h by means of photoelastic study. The addition of 40-60 wt. % filler to composite caused significant increase in hardness, Young's modulus and flexural strength. The *DTS,* after incorporating filler into polymer matrix, was valued at ~26 MPa. The composite containing 40 wt. % of filler demonstrated significantly lower contraction stress in comparison to neat resin and filled > 50 wt. % of silica.

Keywords: experimental dental composite; hardness; DTS; flexural strength; contraction stress.

1 Introduction

Over 70% of all dental restorations are made from composites based on light-curing dimethacrylate resins. These biomaterials replace biological tissue in both appearance and function. Popularity of resin-dental materials is a result of their advantages i.e biocompatibility, application and curing behavior, esthetic and ultimate mechanical properties [1]. Composite dental restorative materials are limited by polymerization shrinkage and its consequence – contraction stress, limited toughness and the possibility of presence of toxic unreacted monomers that remain after unreacted polymerization. Chemistry and structure of polymer matrix, type and amount of filler are the most important aspects determining mechanical properties of dental composites. The most widely used commercial dental resins are prepared *via* polymerization of monomers: bisphenol A glycol dimethacrylate (bis-GMA), urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA), bisphenol A ethoxylated dimethacrylate (bis-EMA) (Figure 1) [2]. An inorganic part of composite are fillers, above all silicon compounds, such as dioxide (amorphous silica, quartz), borosilicates, lithium-, strontium-, as well as barium-aluminum glasses and zirconium and aluminum oxides [3–5].

Resin dental materials have been the focus of a great deal of research with the aim of improving restoration properties in the field of polymerization to decrease stress or improving mechanical features.

In our study, we have concentrated on properties of new experimental dental composites reinforced with different amounts of salinized silica. The filler loading in resin dental composite generally accounts for between 35-70 volume % or 50-85 weight % of composite [6]. The filler has several major roles in ultimate restoration,

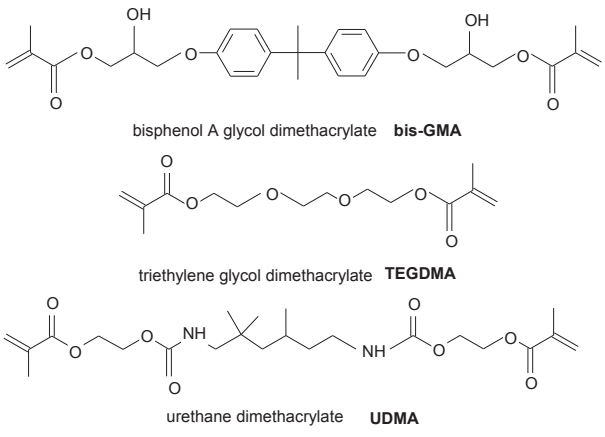

Figure 1: The basic monomers for resin dental composite.

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including enhancing modulus, radiopacity or altering thermal expansion behavior [1,7]. Theoretically, increasing filler content could also minimalize polymerization shrinkage and its consequence contraction stress due to reducing the organic phase volume [8]. Flexural strength and modulus, hardness as well as fracture toughness, are influenced by both filler morphology and filler loading [9]. There is a linear relationship between elastic module and filler loading. There is no clear correlation of fracture strength and fatigue tests to filler fraction, hence materials providing high initial strengths may not reveal the best fatigue resistance [7]. Tanimoto Y. et al, also demonstrated that the bending properties: such as maximum stress and bending modulus, increase with filler content [10].

As mentioned above, the aim of the study was to evaluate the mechanical properties and contraction stress generated during photopolymerization of experimental dental composite in terms of filler concentration.

2 Experimental Procedure

The experimental light-curing composite used in the presented study was developed in the University Laboratory of Material Research at the Medical University of Lodz. The polymer matrix is based on dimetacrylate resins: bis-GMA and TEGDMA (Sigma-Aldrich, USA), 40 - 60 wt. % of precipitated silica filler (Arsil, Z. Ch. Rudniki, Poland) modified with 3-methacryloxypropyltrimethoxysilane (A-174, Sigma-Aldrich, USA) and additions (< 2 wt. %) such as camphorquinone (CQ, Sigma-Aldrich, USA), dimethylaminoethyl methacrylate (DMAEMA, Sigma-Aldrich, USA) and 2,6-di-t-butyl-p-cresol (BHT, Sigma-Aldrich, USA).

The material was cured with Elipar S10 lamp (3M ESPE, Germany) that has an output irradiance of 1450 mW/cm^2 as stated by the manufacturer. To ensure consistent irradiance at each use of the light curing units, the calibrated radiometer system (Digital Light Meter 200 from Rolence Enterprice Inc., Taiwan) was used. The material was irradiated 20 second per 1 mm of material in silicon molds placed between basic laboratory slides. Direct contact of optical fiber with the sample surface was ensured. In order to ensure a satisfactory degree of conversion in the whole volume of material, the polymerization was carried out on both sides of the sample. The methods described below were used to evaluate mechanical properties of cured composites. Samples for this test were prepared by filling the appropriate silicone molds (3 mm high and 6 mm diameter).

Test of diametral tensile strength (*DTS*) is a method used to assess the strength of the dental composite materials according to the specifications ADA 15 (ADA/ANSI Specification 27). Samples were tested using universal testing machine (Z020, Zwick/Röell, Germany). The crosshead speed was 2 mm/min. Diametral tensile strength is the maximum resistance to withstand loads tending to destroy a sample. The numerical value is calculated according to the following formula:

$$\mathrm{DTS\ [MPa]} = \frac{\mathrm{P}}{\mathrm{S}}\frac{\mathrm{[N]}}{\mathrm{[mm^2]}} = \frac{\mathrm{P}}{\frac{1}{2}\left(2\pi\frac{\mathrm{D}}{2}\mathrm{T}\right)} = \frac{2\mathrm{P}}{\pi\mathrm{DT}}\frac{\mathrm{[N]}}{\mathrm{[mm^2]}} \quad (1)$$

where: DTS – diametral tensile strength [MPa], P – load applied [N], S – surface [mm^2], D – diameter of sample [mm], T – thickness of sample [mm].

Semi-automatic hardness tester (ZHμ, Zwick/Röell, Germany) was used to determine the microhardness (HV) of composite materials. The assay was performed according to the Vickers method. Diamond shaped into a square base pyramid with apex angle of 136° is used for testing in this technique. During the test, the indenter was loaded with the weight of 1000 g and the contact time was 10 seconds.

Flexural strength was evaluated in a three-point bending test [11]. The three-point bending flexural test (TFS) was performed using a rectangular specimen (25×2×2 mm), placed on two supports 20 mm apart. The force was applied in the exact middle of the two supports at a 90° angle. Test was conducted according to ISO regulations [12] using universal testing maschine (Zwick Z020, Zwick/Röell, Germany) at crosshead speed of 1 mm/min. The maximum force, at which the fracture of tested material occurred, was measured for each specimen. Flexural strength [MPa] was calculated using following equation [13]:

$$FS = \frac{3Wl}{2bh^2} \quad (2)$$

where: b – width, h – thickness

Photoelastic analysis allows for quantitative measurement and visualization of stress concentration that develops during photopolymerization of resin composites. The method was described extensively in our earlier works [14–16]. Photoelastically sensitive plates of epoxy resin (Epidian 53, Organika-Sarzyna SA, Poland) were used in this study. Calibrated orifices of 3 mm in diameter and thickness of 4 mm were prepared in resin

plates. The diameter of the orifices has been selected to mimic a tooth cavity and clinical conditions of average size. The generated strains in the plates were visualized in a circular transmission polariscope FL200 (Gunt, Germany) and photoelastic strain calculations were based on the Timoshenko equation [17].

Ethical approval: The conducted research is not related to either human or animals use.

3 Result and discussion

Incorporation of filler into resin matrix influences, generally, improves material properties provided that filler particles are bonded to polymer matrix or otherwise, it may act oppositely and weaken the resin. Hence, in our study, we used silanized silica as filler. Benefits of filler content presence are increased hardness, strength, radiopacity and decrease in polymerization shrinkage, thermal expansion and contraction, water sorption, softening, staining and finally improved workability [7].

According to our assumptions and on the basis of Vickers hardness measurements, it has been found that hardness of material surface increases almost 3 times if the filler is incorporated into resin matrix (Table 1). The commercially available composite materials, as delivered by the manufacturer, demonstrated overall significantly higher mechanical properties (DTS, HV) than experimental ones. Composite materials used in dentistry should demonstrate minimum hardness at the level of HV 40–50 [18]. It is crucial, because the aim of a restorative material is to mimic the tissue that it needs to be substituted, namely, enamel and dentine. The average hardness values of dental tissues range from 250 to 360 VHN (Vickers hardness) for enamel and from 50 to 70 KHN (Knoop hardness) for dentin [19]. Most of the conventional dental composites achieve top surface hardness of HV 70–110 [18,20–22], while experimental materials hardness is below HV 40 (Table 1) [23–25]. For our experimental composite material, low hardness (filled has HV = 24÷37, Table 1) narrows its clinical application to lining material or as class V and deciduous teeth restorative material. The commercial low shrinkage composite SDR (*smart dentine replacement*) (Dentsply, Germany) exhibits small DTS and HV values, which can be accepted in clinical conditions if the material surface exposed to occlusion loads (chewing surface) will be covered with a material with better strength parameters [26]. This procedure is recommended by the manufacturer, considering the material is designed as dentine substitute material [7].

Table 1: The effect of filler amount on microhardness of samples (HV) on the cured side to a depth of 1.5 mm of material and polymerization efficiency; photopolymerisation conditions - 20 second/1 mm material thickness, the power of light curing unit 1400 mW/cm^2.

Filler amount, wt. %	HV1		Polymerization efficiency
	polymerization side	1.5 mm depth of material	
0	13 ± 2	11 ± 2	0.87
40	24 ± 4	23 ± 5	0.96
50	30 ± 1	25 ± 3	0.81
60	37 ± 1.5	31 ± 2	0.84

Equally important is the depth of crosslinking of the resin-based composite (DOC) that determines the thickness of the sample for which the optimal properties of the composite are retained. According to ISO standards, the optimal DOC should not be less than 1.5 mm [12]. It is possible to define the depth of crosslinking by dividing the microhardness of the material from the irradiated side to the microhardness on the cross-section of sample [27–29]. The minimum value of this ratio, ensuring that the material of a given thickness will be properly polymerized, is 0.85. It should be emphasized that the lower the value of the coefficient, the greater the amount of residual unreacted monomer, impurities of monomers, additives, degradation products released that can irritate the pulp and soft tooth and perineum tissue, stimulate bacterial growth or promote allergic reactions [30,31]. In our study, not all samples met the ISO standard criterion (DOC = 1.5 mm) (Table 1), concerning the ratio of hardness from the exposed side to the hardness at a depth of 1.5 mm of the sample cross-section, which may translate into the biocompatibility of the material. The decrease of polymerization efficiency in the case of material filled 50 or 60 wt. % is in agreement with Halvorson et al. study, which found that conversion progressively decreased with an increase in filler loading. It could be explained as restricted mobility of resin-monomers due to the existence fillers [32,33]. This led to decreased molecular and radical mobility and resulted in lower monomer conversion.

A significant increase in the Young's modulus is connected to the introduction of filler to the polymer matrix, which increases the rigidity of the composite and reduces its plasticity [7] (Figure 2). Flexural strength for commonly used composite materials range from 60 to 180 MPa [34]. In the case of the presented experimental material, flexural strength exceed 50 MPa (Figure 3). This value meets the ISO standard for materials not suitable with occlusal loads [12] and limits material clinical

indications to restorations in deciduous teeth, class V cavities or lining deep cavities. The obtained results are consistent with our previously published studies [35,36].

Polymerization processes of dental composites generates a shrinkage stress at the restoration-tooth interface (even 20 MPa, dependent on the material) [26]. The consequences of the stress contraction reported in the literature are: deformed tooth structure or even cracks in healthy tooth structure and damage of the adhesive bond between restoration and dental tissue. Shrinkage stresses can also lead to marginal discoloration, post-operative sensitivity and secondary caries [37–39]. Hence, determination of contraction stress is crucial, especially, for new materials. The calculation was conducted following the assumption that the relative change in volume of the composite material causes its extension and extension of the base material which is the "tooth model" (epoxy resin plate). We determined the total state of stress on the surface of the tooth-dentin connection and expressed it quantitatively as reduced stress (d_{int}) (Table 2). On the basis of calculation of the radial stress dr, circumferential stress dq and reduced stress d_{int}, it has been found that the blends containing 40 wt. % of filler have contraction stress reduced by 47% compared to neat resin (Figure 5 and Table 2). The contraction stress of that experimental composite (with 40 wt. % of silanized silica) is the smallest of all made composites. It should be emphasized that the contraction stress on level 7 MPa is, at least satisfactory, considering that low-shrinkage flowable dental composite - SDR has contraction stress account of 5 MPa [26]. The magnitude of the contraction stress has been directly correlated with filler concentration [40,41]. According to linear elastic model theory and Hook's law, the increment in stress is proportional to the increase in materials elastic modulus [42,43]. This is in line with our Young's modulus measurements (Figure 2). Material with 40 wt. % of filler exhibited elastic modulus of 2 GPa while composite with 50 or 60 wt. % of filler exhibited E ~ 3.3 GPa (Figure 2). Such low contraction stress could also be explained due to the possibility of occurrence of viscous flow. The plastic flow, mostly occurring prior to the acquisition of significant elastic modulus, allows part of the shrinkage to take place without stress built up at the interface and leads to remaining contraction stress reduce (Figure 5 and Table 2).

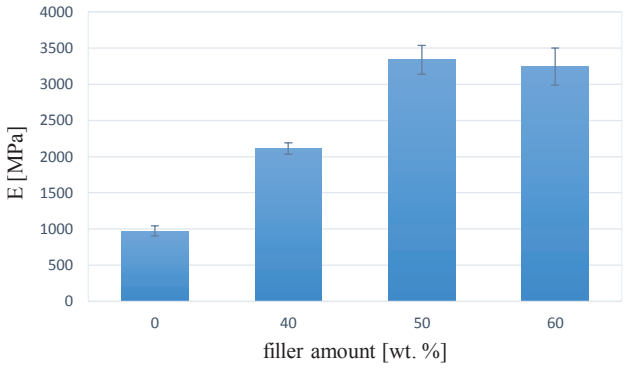

Figure 2: The influence of filler amount on Young's modulus (E) of neat resin and composite filled with 40, 50 or 60 wt. % silanized silica; photopolymerization conditions - 20 second/1 mm material thickness, the power of light curing unit 1400 mW/cm².

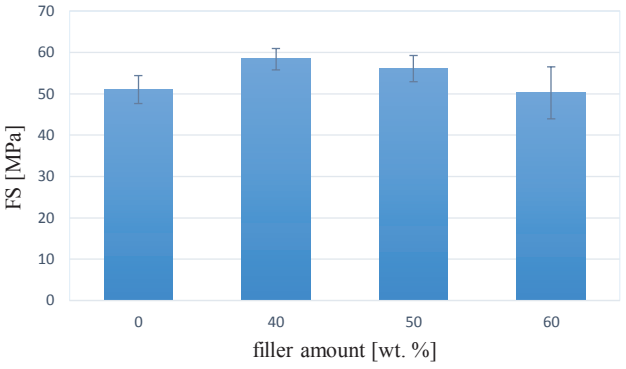

Figure 3: The influence of filler amount on flexural strength (FS) of neat resin and composite filled with 40, 50 or 60 wt. % silanized silica; photopolymerization conditions - 20 second/1 mm material thickness, the power of light curing unit 1400 mW/cm².

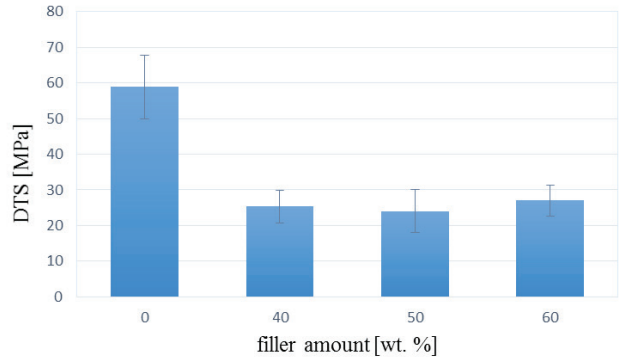

Figure 4: The influence of filler amount on diametral tensile strength (DTS) of neat resin and composite filled with 40, 50 or 60 wt. % silanized silica; photopolymerization conditions - 20 second/1 mm material thickness, the power of light curing unit 1400 mW/cm².

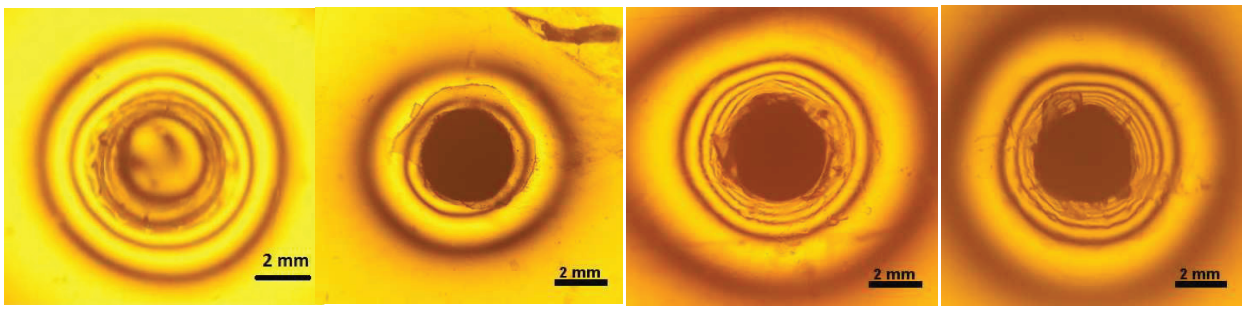

Figure 5: Isochromes in epoxy resin of experimental material with and without filler (0 ÷ 60 wt.%) around restorations, acquired in polarized light with parallel polarization facets.

Table 2: The influence of filler amount on the radial stress (d_r), circumferential stress (d_q) and reduced stress (d_{int}) generated during polymerization of experimental dental composite of neat resin and composite filled with 40, 50 or 60 wt. % sialnized silica; photopolymerization conditions - 20 second/1 mm material thickness, the power of light curing unit 1400 mW/cm^2.

Filler amount, wt. %	d_r, MPa	d_q, MPa	d_{int}, MPa
0	5.8 ± 0,2	-6.7 ± 0.2	12.5 ± 0.2
40	2.7 ± 0,2	-3.9 ± 0.2	6.6 ± 0.1
50	7.9 ± 1	-9.3 ± 1.2	17.2 ± 2.2
60	9.2 ± 1	-11.1 ± 1.2	20.3 ± 2.2

4 Conclusions

The incorporation of silanized silica as filler to the experimental dental composite influenced significantly its properties. The hardness and elastic modulus increased with the amount of filler. The flexural strength and diametral tensile strength remained on the same level regardless filler concentration. Commercially available composite material showed higher mechanical properties than the experimental ones. Still, experimental material meets the ISO standard for materials used as suitable to restorations in deciduous teeth, class V cavities or lining deep cavities.

Considering the negative impact of contraction stress on clinical performance of bonded restoration, it needs to be emphasis that the experimental composite (bis-GMA/TEGDMA/40 wt. % silanized silica) has low stress values. Conflict of interest: Authors state no conflict of interest.

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