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Original research article

## Improvements in self-curing composites

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## ABSTRACT

**Purpose:** The purpose of this study was to test the influence of a barbituric acid derivative acting as a catalyst and small amounts of pyrolytic silica in acrylic resins on color stability, solubility and sorption of a composite.

**Materials and methods:** A series of two-component powder/liquid resin systems were prepared. Monomer-like mixtures (bis-GMA, TEGDMA, tertiary amine 60/40) and a quartz powder with additions of various silica and barbituric acid derivatives were used. Temperature of the material during polymerization was measured with the use of a thermometer. In addition, the material's flexural and compressive strength, sorption and solubility were tested pursuant to ISO4049:2009.

**Results:** The powder-based acrylic composition in a liquid mixed immediately before use, after an addition of a 0.5% barbituric acid derivative, has a lower temperature during the polymerization process (a reduction from 43 °C to 37 °C), whereas color stability over time is improved, with  $\Delta E = 1.81$  for samples of powder mixtures containing between 0.45% of BPO and 0.15% of barbituric acid derivatives. For silanized quartz powder with 0.55% BPO and 0.1% BA + 0.5% Aerosil R711, the obtained sorption value was  $4.57 \pm 0.22 \mu\text{g}/\text{mm}^3$ , whereas solubility was  $1.60 \pm 0.32 \mu\text{g}/\text{mm}^3$ .

**Conclusions:** New catalytic system with barbituric acid derivative, improves color stability for samples stored at room condition and under light of high intensity. A two-phase composite (bis GMA TEGDMA/ Quartz), with a new catalytic system with barbituric acid derivatives, has a lower self-cured temperature. Adding a small quantity of hydrophobic silica (0.5%) has a significant influence, with reduced sorption and solubility of the material.

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## 1. Introduction

Self-cured and light-cured adhesive composite resins have their biological advantages and disadvantages. Acrylic resins polymerizable at room temperature are used in many applications in dental technology [1–4]. They serve, among others, to repair dentures and in the pouring technique (powder and liquid are mixed and poured into agar or silicone molds). In dental offices, self-curing, bis-acrylic, and composite resins are used for core build-ups, or in temporary crowns and bridges. Composite materials that polymerize at low temperatures are mostly based on a redox catalytic system peroxide, i.e. a tertiary amine. This system has a number of drawbacks, among which there is the quick color change to yellow over time, which obviously influences the clinical use of this type of material. Another disadvantage is the high temperature during the polymerization process, which may reach 60 °C, causing an

increase in temperature in the pulp chamber of the tooth, resulting in damage or necrosis [3]. To overcome the problem of high temperatures during polymerization, a new curing system based on barbituric acid derivatives was designed 15 years ago [5–7].

Literature provides information on barbituric acid derivatives, especially 1- benzyl- 5 phenyl barbituric acid (BA) [6,7], a substance that can generate free radicals necessary for starting polymerization of double bond in methacrylic resins. Additionally, the material contains a heterocyclic ring with nitrogen atoms (Isocyanurate group), which can positively influence color stability. In literature it is possible to find information on barbituric acid derivatives used as a catalyst for self-curing temporary crown and bridge materials in cartridge form with mixing ratios of 10:1 or 4:1 (Prothemp 3 3MESPE). The first patent for barbituric acid derivatives as a catalyst dates back to 1978.

Within the last 20 years improvements in dental composites have been made, brought by the use of fine nano-scale particles. Nano materials are very popular in various industries, e.g. fumed silica is used in dental technology in many light-cured composites for improving their mechanical and rheological properties. Balos and al. describe improvements in flowable composites after small

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amounts (<1%) of hydrophobic nano-silica particles are added [8]. Khaje described improvements in the mechanical properties of a light-cured material resulting from the use of higher amounts of fumed silica [9].

## 2. Materials and methods

The purpose of this study was to improve color stability and mechanical properties of self-cured composite resins. A model powder/liquid system was chosen on the basis of the commercial product available on the market called Evicrol (Spofa Dental). The powder was quartz containing 1.5–2.0% of Benzoyl Peroxide (BPO) as a catalyst. Pigments based on natural oxides were added to the powder to imitate tooth color. The liquid were bis-GMA and a triethylene glycol dimethacrylate (TEGDMA) 50-50% mixture with Tinuvin P as a light stabilizer and *N,N* di hydroxyl ethyl p toluidine as a catalyst. The liquid sample was donated by Spofa Dental. After mixing the powder with the liquid, the obtained material was placed in the oral cavity and cured for 1 min in 37 °C. This reaction would have taken longer in a lower temperature. After a short period of time, the material changed its color to yellow. This process resulted from higher concentrations of benzoyl peroxide and amine [10].

### 2.1. Sample preparation

Salinized quartz obtained from Spofa Dental was mixed with appropriate amounts of benzoyl peroxide (Sigma Aldrich) and 1 benzyl- *p*-phenyl barbituric acid (Chemishe Fabric Berg). The mixture of quartz and benzoyl peroxide was prepared in a ball mill with a capacity of 2 liters (Fritch), with a speed of 40 rpm, and a mixing time of 120 min. Finally, a barbituric acid derivative was added, after which the substance was mixed for 5 min. Eight different powder compositions were prepared in a quantity of 100 g each (Table 1).

In order to reduce sorption and solubility, different kinds of fumed silica nanoparticles were added to other samples in a concentration of 0.5%, i.e. Aerosil R 812 hydrophobic silica (Evonik), HDK 2000 (Waker), and Aerosil R711 (Evonik). The disadvantage of raw materials of this kind is their very small density. For this reason it is possible to add approx. 0.5% of such material to the composition, due to its mixing ratio with liquid. The powder comprising sample number 6 was a mixture of Silanized quartz and Kavitan glass ionomer powder (Spofa Dental).

### 2.2. Methods

#### 2.2.1. Temperature measured after mixing of powder and liquid

During the setting, the reaction itself, and the breaking of the double bond, a great amount of heat energy is emitted. Too high temperatures can be dangerous for tooth pulp, causing its necrosis. For this reason, all the compositions of self-cured resins were tested through temperature measurement during setting time. For

the tests, the liquid and powder were mixed for 40 s with the use of a balance (Denver Instruments Xp-3300). A stopwatch (JPD System) was activated for the same period. In each of the tests of the first series of experiments, 0.5 g of Evicrol liquid was mixed with 1.5 g of powder. After obtaining a homogeneous paste material, it was placed in polyethylene cups and a thermometer (Multi thermos 300) was immersed inside the mass. The tip of the thermometer was covered with aluminum foil to protect against the mass adhering after setting. Each 15 s the temperature was measured and recorded on a piece of paper. The end of polymerization was established as the moment when the temperature was the highest. Each test was repeated 3 times.

#### 2.2.2. Compressive strength

Six samples of each of the compositions (a total of 48 samples) were tested on a Shimadzu (AGS 10 kNG) compressive strength instrument. The dimensions of the samples were chosen pursuant to ISO 4049 (Dental filling materials), i.e. a diameter of 6 mm and a height of 20 mm. The tests were carried out 24 h after the samples, stored at room temperature, were prepared. The rate of the head breaking in the three-point bending test was 0.25 mm/min. The end of the test was established as the moment when the sample was destroyed.

#### 2.2.3. Flexural strength

Six samples of each of the compositions (a total of 48 samples) were tested on a Shimadzu (AGS 10 kNG) flexural strength instrument. The dimensions of the sample were chosen pursuant to ISO 4049 (Dental filling materials) [11,12], i.e. 2 × 2 × 40 mm. The tests were carried out 24 h after the samples, stored in room temperature before the test, were prepared. The end of the test was established as the moment when the sample was destroyed.

#### 2.2.4. Sorption and solubility

Five samples of each of the compositions (a total of 35 samples) were tested [12]. The mass of each of the samples was measured before commencing the test with the use of an analytical balance (Denver Instruments Xp-3300). The dimensions of the samples were: thickness of 1 mm and diameter of 10 mm. After the measurements, the samples were stored in an exactor with a silica drying gel to obtain a constant mass (M1). Then the samples were placed in water (37 °C) for 1 week. After this time the material was removed, dried with paper, and weighed again, to obtain the second value (M2). In the next step, each sample was dried in an exactor at 37 °C to obtain a constant mass called (M3). Solubility was calculated according to the following equation:  $R = (M1 - M3) / M1$ . Sorption was calculated according to the following equation:  $S = (M2 - M1) / M1$ .

#### 2.2.5. Color change

Colors of 10 samples (a total of 70 samples) from each of the tested groups were measured in a colorimeter (eXact™ X Rite) employing an optical geometry of 45°/0°. The color was

**Table 1**  
Compositions of different powders used in the tests.

Number	Composition
1	Silanized quartz powder with 2% BPO as a background sample
2	Quartz powder with 0.9% BPO and 0.08% BA
3	Silanized quartz powder with 0.45% BPO and 0.15% BA
4	Silanized quartz powder with 0.4% BPO and 0.1% BA
5	Silanized quartz powder with 0.55% BPO and 0.1% BA + Aerosil R 812
6	Salinized quartz powder with 0.55% BPO and 0.1% BA + 50% glass Kavitan LC
7	Silanized quartz powder with 0.55% BPO and 0.1% BA + 0.5% HDK (surface treated silica Waker)
8	Silanized quartz powder with 0.55% BPO and 0.1% BA + 0.5% Aerosil R 711 (surface treated silica Evonik)

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