

ARTICLES

CHARACTERIZATION OF LIGHT-CURED DENTAL COMPOSITES PREPARED FROM BIS-GMA/TEEGDMA AND BIS-GMA/ MPS MIXTURES

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Abstract— The aim of this study was to prepare light-cured dental composites and to evaluate their physical, chemical and mechanical properties. Experimental composites were prepared from 2, 2 bis [4-(2-hidroxy 3-metacriloxypoxi) phenyl] propane (Bis-GMA), tetraethyleneglycol dimethacrylate (TEGDMA), 3-methacryloxypropyl trimethoxysilane (MPS) and a hybrid filler consisting of quartz and colloidal silica particles (Aerosil 90). The initiation system was based on the pair Camphorquinone (CQ)/N, N dimethylaminoethylmethacrylate (DMAEMA). The experimental composites and a commercial dental composite used as reference (Charisma[®]) were submitted to determinations of depth of cure, water absorption and solubility, compressive and diametral tensile strengths, and elastic modulus, according to the indications of the ISO standards. The experimental resins presented adequate values for most of the analyzed properties, except for compressive strengths. In the Bis-GMA/ MPS/quartz-aerosil composite a better integration between filler and matrix was observed. The Bis-GMA/TEGDMA/quartz-aerosil composite showed the best performance in regards to depth of cure, solubility in water, diametral tensile strength and elastic modulus. The properties of this composite were better than those of the commercial reference material, except for compressive strength.

Keywords— light-cured, dental composites, depth of cure, absorption, solubility, mechanical properties

I. INTRODUCTION

During many years dental amalgam was the ideal restoring material for the damaged dental tissue, due to its high mechanical strength and durability, but it lacks of aesthetic and mimetic with the dental tissue. (López-Amo, 2001; Garro, 2001). The dental composites or compound resins appeared as an aesthetic and mimetic alternative to dental amalgam. They consist of two main components, the organic matrix and the inorganic filler. The organic matrix is formed by dimethacrylates, which are non-toxic and capable of a rapid free-radical polymerization, even in the presence of oxygen and water. The unfilled matrix has a poor wear resistance by itself, which can be improved by the inclusion of particulate

fillers, which are harder than the polymeric matrix (Kalachandra *et al.*, 1997; Sideridou *et al.*, 2002).

For dental restorative composites the monomeric mixture commonly consist of Bis-GMA and a viscosity modifier (diluent) as triethyleneglycol dimethacrylate (TEGDMA), 3-hydroxypropyl methacrylate (HPMA), TEGDMA and MPS (Vehoven *et al.*, 1994; Habsuda *et al.*, 2002).

The polymerization of dimethacrylates can be chemically or visible light-initiated, but light-initiation is preferred because it allows a better control of the entire polymerization process. Thus initiation can be started and stopped almost at will.

A basic mechanical property of a restorative material, such as light-cured resin composites, usually presented by manufacturers, is its strength. Data usually provided includes compressive, tensile, or flexural strengths. These strength values are usually related to filler content and composition or to different curing times (conversion degree of the matrix) and packing procedures (Brosh *et al.*, 1999).

Composites are classified according to their viscosity (flowable or condensable) and also according to the type and size of the fillers (macrofillers: 10-30 μm , microfillers: 0.01-0.04 μm and hybrid fillers: mixture of macrofillers and microfillers).

The use of inorganic fillers consisting of rational mixtures of fractions with different particle size increase packing density and filler content, improve the strength, ensures that the material can be easily polished and decrease the polymerization shrinkage of the resulting composite. Thus, these hybrid composites can be used for dental restorations in posterior teeth where masticatory forces are particularly intense (Leylanaz and Santerre, 1999; Davidenko *et al.*, 2001; Paik *et al.*, 2001).

The water absorption of dental resins, either filled or unfilled, is important for dental applications. In filled dental resins the absorbed water decrease the life of the restorations by hydrolysis of the bonds developed between the filler surface and the coupling agent with results in cracks formation. The water ingress may have beneficial effects concerning the expansion of the composite, compensating thus the polymerization shrinkage with improved marginal sealing and relaxation of the stresses set up within the matrix during shrinkage (Mar-

tin and Jedynekiewicz, 1998; Santos *et al.*, 2002; Sideridou *et al.*, 2003, 2004).

The polymer solubility is related to the amount of unreacted monomer leachable by water. The water is trapped during the polymerization inside the microgels between the polymer chains and it is adsorbed to the surrounding network, or it is trapped in the micropores. (Sideridou *et al.*, 2003).

Silanes coupling agents has been predominantly employed since the early 1960s, as bonders at the resin-filler interface. Classically, the coupling procedure is carried out in two steps. In the first step the filler is treated with the silane coupling agent, usually MPS, and at the second step the treated filler is blended with the monomers mixture to form the pre-cured composite paste. An alternative coupling method is the use of MPS at higher concentrations as a substitute of the diluents in the monomer mixture in one-step silanization procedure. The Bis-GMA/MPS mixture offers the opportunity for silane coupling to fillers because of the presence of the trimethoxysilyl group in MPS. The resins prepared with Bis-GMA/MPS mixture showed adequate results for some properties, such as polymerization contraction, conversion and wear strength (Vehoven *et al.*, 1994; 1996). However, no reports have been found on the depth of cure, water absorption and solubility, compressive and diametral tensile strength, and elastic modulus of dental resins based on the monomers Bis-GMA/MPS.

The aim of this work was to prepare two light-cured dental resins; using the mixtures Bis-GMA/TEEGDMA and Bis-GMA/MPS as organic matrixes and hybrid filler consisting of quartz and Aerosil 90, and to compare them with a commercial dental composite with regards to depth of cure, water absorption and solubility, compressive and diametral tensile strengths and elastic modulus.

II. MATERIALS AND METHODS

A. Preparation of composites

Aerosil 90 (0.04 μm , colloidal silica, provided by Degussa) and Cuban quartz ($30 \mu\text{m}$, supplied by Laboratory "José Isacc del Corral"), both with purity > 99.5%, were used. The particle morphology for both fillers and fracture surface of composites were analyzed by (SEM) scanning electron microscopy (JEOL 6400 Microscope-LINK AN 10000).

The organic matrix consisted of 60 wt% Bis-GMA, and 40 wt% diluent comonomer. The diluents comonomers were TEEGDMA and MPS. The initiation system was based on the pair CQ / DMAEMA (0.5:0.5 wt %). Diluents, initiator and coinitiator were supplied by Fluka.

Charisma®, Heraeus Kulzer, was used as commercial reference material. Table I shows the composition of the studied resins.

The dental composites and unfilled resins were cured with a halogen lamp Visilux II for 40 s and 20 s, respectively.

B. Depth of cure test

The depth of cure of composites was determined according to the ISO 4049 (2000) standards and 6874 (1988) for unfilled resins. The composites were filled into a stainless steel moulds (4 mm diameter x 6 mm long) covered with transparent strips. The excess material is extruded by squeezing it between two microscope slides and cured for 40 s from the top of the mould. Immediately after completing irradiation, remove the specimen from the mould and remove the uncured material with the plastics spatula. The height of cured specimen was measured with a micrometer (0-25mm, accuracy 0.01 mm), this value was divided by two and it was reported as the depth of cure of the material. Three sample of each composite were evaluated.

Table I. Composition of dental resins

System	Monomeric Mixture		Filler	
	Composition (wt %: wt %)	wt %	Quartz (wt %)	Aerosil 90 (wt %)
MM _I	Bis-GMA/TEEGDMA (60:40)	100	-	-
MM _{II}	Bis-GMA/MPS (60:40)	100	-	-
RC _I	Bis-GMA/TEEGDMA (60:40)	23.00	73.00	4.00
RC _{II}	Bis-GMA/MPS (60:40)	19.04	77.65	3.31
COM	Bis-GMA/TEGDMA	21	79 % Barium-alumino-boro-silicate glass/silicon dioxide	

C. Absorption and solubility in water tests

Water sorption and solubility tests were determined according to the method described in ISO 4049 (2000) standards norm. Five cylindrical specimens (15 mm diameter x 1 mm thickness) were prepared into Teflon mould between two glass slides covered and cured accordance with the procedure specified in ISO 4049 (2000) standards. On removal the specimen were placed in a desiccators containing freshly dried silica gel. After 22 h they were removed, stored in desiccators at 23°C for 1 h and weighed with a precision of 0.01 mg. This cycle was repeated until a constant mass (m_1) was obtained. The discs were immersed in distilled water at 37°C for seven days, then removed, blotted dry and weighed (m_2). After this weighing, the specimens were reconditioned to a constant mass (m_3) in the desiccators using the above described cycle. The thickness and diameter of the samples were measured accurately at five points using a micrometer (0.25 mm) and using these measurements their volume was calculated in mm^3 .

The values of water absorption (W) and solubility (S) were calculated for each disc using the Eqs. 1 and 2, respectively.

$$W = m_2 - m_3 / V \quad (1)$$

$$S = m_1 - m_3 / V \quad (2)$$

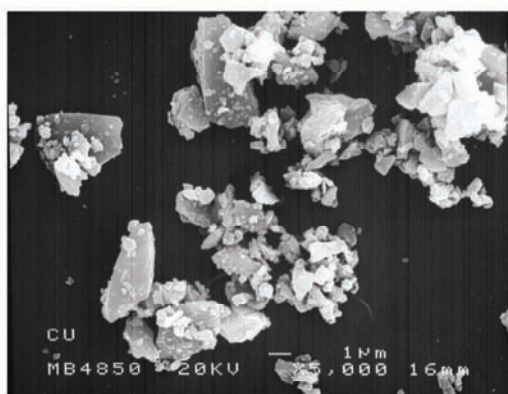


Figure 1. SEM micrograph of quartz.

D. Compressive strength test (C)

The mechanical properties were determined according to the ISO 604 (1997) standard.

Ten cylindrical specimens (4 mm diameter x 8 mm height) were prepared by packing the resin composites into Teflon molds. Specimens were cured for 40 s from each end and stored in water at 37 °C for seven days prior to testing. A testing machine MTS Bionix 858 was used at a crosshead speed of 1mm/min. Compressive strength was calculated by dividing the failure load (F_m) by the cross-sectional area.

E. Diametrical tensile strength test (DTS)

Ten cylindrical specimens (6 mm diameter x 4 mm height) were prepared using Teflon molds. The fabrication, storing and loading of specimens procedures were the same employed in the compressive strength test. The diametral tensile strength was calculated using the Eq.3.

$$DTS = 2F/\pi Dh \quad (3)$$

F. Statistical procedure

For each test the mean value and standard deviation were calculated and compared using the Student Test and ANOVA at a significance level of 0.05 %.

III. RESULTS AND DISCUSSION**A. Characterization of the fillers**

The current state of the art in dental composites recommends the use of a mixture of micro and macro particles to obtain hybrid filler. In this work a mixture of quartz (macroparticles) and Aerosil 90 (microparticles) was employed to achieve good mechanical properties and polishing capability. The shape of the particles is another important aspect influencing the mechanical locking of filler particles to the polymeric matrix; an irregular shape of the filler particle favors a better physical retention in the polymeric matrix, however, irregular particles possesses smaller packing ability and therefore they cause an heterogeneous stress distribution. Figures 1 and 2 show the morphology and size of particles of fillers.

The irregular shape observed for the quartz crystals (Fig. 1) coincides with other reports for this filler. On

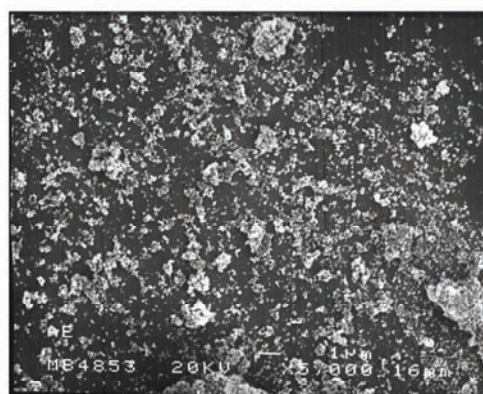


Figure 2. SEM micrograph of Aerosil 90.

the other hand it was observed a wide particle size distribution, ranging 1-15 µm, also previously observed (Frómeta, 1998).

In the case of Aerosil 90 (Fig. 2), described as pyrogenic silica by the manufacturer, aggregates of spherical nanometric particles was observed. A filler with this characteristics should improve the workability of the uncured composite and the capability to obtain a smooth surface by polishing in the final composite, however, if it is used alone without compounding with a macro-filler, the amount admissible by the monomeric matrix is very small (around 30wt.-%) and the mechanical properties would be low.

B. Preparation of composites

Both unfilled resins were yellowish, due to the CQ. The MM_{II} unfilled resin is less viscous than MM_I.

The two composites were homogeneous pastes and they had similar coloration. The RC_{II} composite is the most viscous and hardest to work. The reference resin was a homogeneous paste, beige colored, and lightly more viscous that RC_I and it is easy to work.

The smallest content of filler, 77 wt.-%, corresponds to the RC_I composite, while in the RC_{II} it was possible to reach 81 wt.-%, due to lower viscosity of the MPS in comparison to TEEGDMA. The filler wettability also should be favored in the presence of MPS, which is able to chemically interact with silanol groups at the filler surface and to lower the polarity of the filler surface, making it more compatible towards the monomers mixture.

C. Depth of cure

The depth of cure (D_c) is an important property of the light-cured dental resins. It is a measure of the polymerization efficiency, decisive aspect in the composites performance. Mechanical properties such as flexural strength, strength, and modulus of elasticity depend of the conversion degree of the matrix. Other properties such as color stability, solubility and degradation are strongly related with the degree of cure (Eliades and Brantley, 1995).

Table II shows the results of depth of cure for the filled and unfilled resins.

When comparing the MM_I (2.89 mm) and MM_{II} (2.86 mm) systems that correspond to the polymeric matrix, it was observed that both fulfill the requirement of the ISO 6874 (1988) standard since the obtained values were higher than 1.5 mm. They showed significant differences.

The MM_I unfilled resin showed the higher value of this property, due to the presence of TEEGDMA as diluent, which is a bifunctional monomer able to polymerize through both ends of the molecule, and permits a higher degree of crosslinking, smaller amount of residual double bonds and a higher efficiency in the polymerization.

Table II. Depth of cure (mm) of resin composites

Classification	Systems	Dc ± Sd
Bonding	MM _I	2.89 ± 0.01
	MM _{II}	2.86 ± 0.01
Composites	RC _I	2.74 ± 0.01
	RC _{II}	2.13 ± 0.04
	COM	2.46 ± 0.01

On the other hand, the obtained values of depth of cure for all the composites were superior to the value of 2 mm required by the ISO 4049 (2000) standard. The RC_I resin was the only of them that exhibits a loss of uncured material (0.26 mm) lower than 0.5 mm as required by ISO 4049 (2000). The other two systems do not fulfill this specification, although the commercial resin (COM) presents a value near to specified one. Significant differences exist among the results of depth of cure obtained for the three composites. The RC_I composite presents a depth of cure higher than RC_{II} and commercial resin. Degree of conversion at 30s of the experimental resins measurement photocalorimetric indicates that RC_I (45 %) had higher conversion than RC_{II} (36 %) (Davidenko *et al.*, 2003).

The RC_{II} composite, containing MPS in its organic matrix, showed the lowest value of depth of cure. The polymer network in this composite has the lowest degree of crosslinking and higher amount of unreacted monomer, due to mono functional character of MPS. It was also the composite that has the highest filler content. When comparing the results (Table II) for the unfilled resins MM_I, MM_{II}, with the RC_I and RC_{II} composites, respectively, it was observed that the unfilled resins present values of depth of cure significantly higher than those of the related composites (MM_I and RC_I, MM_{II} and RC_{II}), due to the decreasing of molecular mobility at the filler-matrix interface and the absorption and/or dispersion of the light at the filler particles during irradiation (Davidenko *et al.*, 2001).

D. Water absorption and solubility

The water absorption (W) and the solubility (S), are parameters that should be controlled in the dental composites. In the oral environment dental composites may absorb water and release unreacted monomers, initia-

tors, catalysts, stabilizers, etc, soluble in the aqueous media. The release of soluble components from resin composite may stimulate the growth of bacteria around the restoration (Hansel *et al.*, 1998) and to promote allergic reactions in some patients (Spahl *et al.*, 1994; Sideridou *et al.*, 2003).

Figure 3 shows the results of water absorption and solubility of bonding, composites and commercial resins studied.

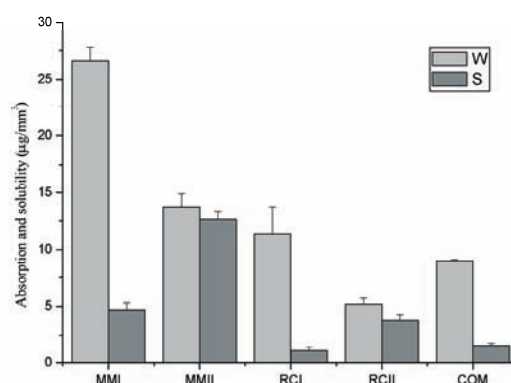


Figure 3. Water absorption and solubility ($\mu\text{g}/\text{mm}^3$).

These properties in the composites obey the same tendency, but their values were lower than in unfilled resins. The lower water absorptions and solubility of composites is due to the lack of absorption capacity and insolubility of the fillers. However, it can not be discarded the contribution of the filler-matrix interface that presents silane condensed silanol groups able to hydrolyze and form hydrogen bonds with water that favor the absorption, and the hydrolyzed silane can be extracted in water contributing to solubility.

According to ISO 4049 (2000) standard a dental restorative resins, in order to be suitable for clinical use, must absorb less than $40 \mu\text{g}/\text{mm}^3$ of water and loss by solubility less than $7.5 \mu\text{g}/\text{mm}^3$. All studied composites and the unfilled resin, MM_I, fulfill this standard. The unfilled resin MM_{II} fulfills the requirement of absorption but its solubility exceeds the limit of ISO standard.

The MM_I system showed higher absorption ($26.6 \mu\text{g}/\text{mm}^3$) and lower solubility ($4.7 \mu\text{g}/\text{mm}^3$) than MM_{II} ($13.7 \mu\text{g}/\text{mm}^3$, $12.6 \mu\text{g}/\text{mm}^3$, respectively). The absorption values for Bis-GMA/TEEGDMA systems were much higher than the corresponding to Bis-GMA/MPS system because TEEGDMA units are more hydrophilic than MPS ones. The greater solubility of the system based on Bis-GMA/MPS is caused by its lower cross linking and conversion as already explained above. RC_I did not differ significantly of the commercial resin with regards to water absorption and solubility (11.4 vs 9.0 and 1.1 vs $1.5 \mu\text{g}/\text{mm}^3$, respectively).

The higher solubility in MM_{II} and RC_{II}, is mainly due to the use of MPS in their formulations. On the other hand, the results agree with those obtained for the depth of cure which behave inversely, i.e., the smaller the conversion, the higher the amount of free monomer able to be extracted by water. Besides, it should be kept

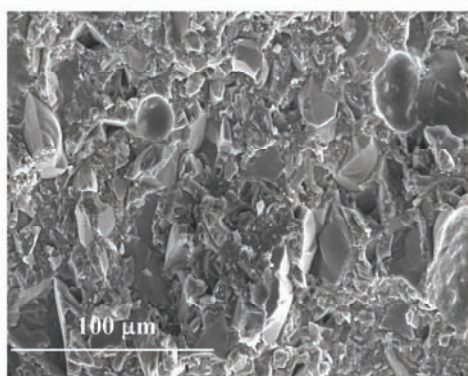


Figure 4. Fracture surface of Bis-GMA/ TEEGDMA/ quartz aerosil composite.

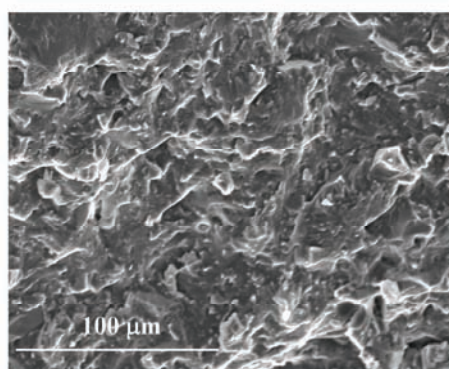


Figure 5. Fracture surface of Bis-GMA/ MPS/ quartz aerosil composite.

in mind that the filler treated that should take place when mixing the filler and the monomers mixture produces methanol, which is also extractable, and contributes to create some extra porosity in the composite. Porosity represents a disadvantage towards the mechanical properties of the material, but it might be beneficial in reduction of stresses caused by polymerization contraction (Alster *et al.*, 1992; Vehoven *et al.*, 1994).

E. Mechanical properties

Dental composites are designed to perform under cyclic mechanical stress caused by mastication forces, so their mechanical properties allow predicting their *in vivo* behavior. Some of the mechanical properties, such as wear strength, are sensitive to the particle size of the filler. On the other hand, hardness and compressive strength depend more on the filler content than on its particle size. Table III shows the results of compressive strength (C), diametral tensile strength (DTS) and modulus of elasticity, obtained for the experimental composites and the commercial resin.

Table III. Results of mechanical tests

Resin	C ± Sd (MPa)	E ± Sd (GPa)	DTS ± Sd (MPa)
RC _I	143 ± 20*	39 ± 12*	35 ± 2
RC _{II}	121 ± 22*	25 ± 9	26 ± 4
COM	280 ± 66	30 ± 15*	44 ± 7

*There is not significant differences

A dental composite should be at least as strong as the tissue it substitutes. The minimum recommended values of compressive and diametral tensile strengths are 200 and 30 MPa, respectively.

Light-cured resin composites are characterized by high compressive and low tensile strengths. The importance of tensile properties of resin composites has been recently emphasized; however, the relationship between these two strength values has not been extensively studied (Brosh *et al.*, 1999).

The RC_I resin exhibited the best compressive strength, but it was not significantly different of the ob-

tained for RC_{II}. The slight difference could be attributed to the presence of MPS in the matrix RC_I and its coupling action that probably improves the matrix-filler bonding. The figures 4 and 5 show the fracture surfaces obtained by SEM of composites RC_I and RC_{II}, respectively. In those composite that contains the silane (RC_{II}) a better integration between filler and matrix was observed.

RC_I and RC_{II} had lesser compressive strength than the resin Charisma®, probably due to pores caused by air inclusion during the pastes and probes preparation. Also, in commercial resin the filler was previously treated favoring a better interaction filler- matrix.

The diametral tensile strength of RC_I was significantly higher than that of RC_{II} and satisfied the minimum strength required (30 MPa). However, it was lower than that of the commercial resin.

The moduli of elasticity (Table III and Fig. 6) of all the resins were slightly higher than that of dentin (18 GPa). According to the elastic modulus the most appropriate restorative material should be RC_{II}, due to its modulus almost matches the dentin's. RC_I presented the higher modulus, what indicates it is the most rigid of all. The commercial resin was not significantly different of RC_I, but did from RC_{II}, with regards to the elastic modulus. All the resins exhibited elastic moduli smaller than enamel, which favors the resin performance according to some authors (Brosh *et al.*, 1999; Diaz *et al.*, 1999).

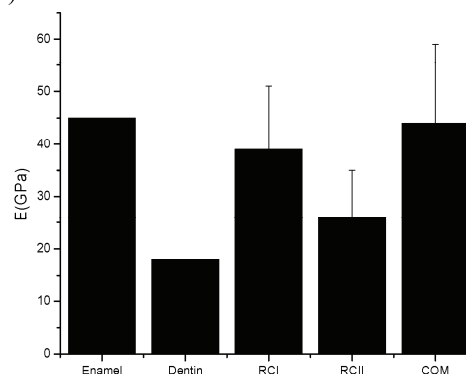


Figure 6. Modulus of elasticity of enamel, dentin and composites studied.

In general, the experimental resins presented acceptable values for most of the analyzed properties. Although they do not fulfill the requirement of compressive strength, they are similar, and occasionally better than the commercial resin with regard to the remaining properties.

The RC₁ resin exhibited the best global performance, even when its compression strength is not very high; and could be indicated for restorations in anterior teeth. Its compressive strength could also be improved by using silane-treated filler to achieve a better filler-matrix bonding. Excluding air inclusion during pastes preparation should provide an additional improving of the strength.

IV. CONCLUSIONS

The experimental resins presented adequate values for most of the studied properties, except for compressive strengths. In the Bis-GMA/ MPS/quartz-aerosil composite a better integration between filler and matrix was observed. It showed adequate values water absorption, elastic modulus and diametral tensile strength.

The Bis-GMA/TEEGDMA/quartz-aerosil composite showed higher values of depth of cure, water absorption, modulus of elasticity and lower solubility and compressive strength than commercial composite. Diametral tensile strength was similar for both resins.

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