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THE INFLUENCE OF THE REINFORCING MATERIALS ON THE PROPERTIES OF THE DENTAL COMPOSITES

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ABSTRACT. The influence of inorganic reinforcing materials and their mixtures on the strength, hardness, and color shade of composite materials was studied. Variation of grain size distribution and specific area on composite surface appearance and on tensile and compressive strength was also studied. The size and the shape of the particles were investigated with a TESLA BS-500 electronic microscope. The particles have irregular shapes. Grain size distribution was determined with a COULTER-COUDLTER instrument, and specific area corresponding to each grain size was determined by the BET method. Compressive and tensile strength were determined with an INSTRON device. The best results regarding compressive strength were obtained using powders with specific area of 2.1 m^2/g. Abrasion tests were made after maintaining the samples at 37°C in water for a week. Rate of wear was also investigated. The rate of wear increases as the specific area increasing from 1.9 to 2.9 m^2/g. The obtained results are between imposed limits for dental materials.

INTRODUCTION

The literature offers a series of examples of inorganic reinforcing materials used as reinforcing materials for dental composites: silicates, lithium aluminosilicates[1-4], hidroxylapatita[6,8], glasses[5,7,8], quartz[1,-5,7], silica[1-9], ceramics[5,10] and oxides (Al_2O_3, TiO_2)[11]. From those materials, silica and quartz have been successfully used for composites used in dentistry[4-9]. It is important that those materials to be *chemical inert* for ensuring the resistance of the composite at the action of the agents from the mouth cavity. The coefficient of thermal expansion of the composite mast be as close as possible to those of hard dental tissue (α=10-11·10^{-6}/°C). The refractive index closed to the one of the enamel offers to the composite esthetical properties. Good mechanical strength for improving the mechanical properties of the composite must be close to those of the hard dental tissue. Taking into account those facts the composites with quartz and colloidal silica[5,7] are suitable for this kind of applications.

The aim of this paper is the characterization of the inorganic reinforcing materials used for obtaining of dental composite, as well as their influence on the mechanical proprieties of those.

EXPERIMENTAL DETAILS

Materials and methods. The technical characteristics of quartz and silica were studied in comparison to the ones of the inorganic phases from enamel and dentine.

Quartz powder. Quartz powders of different grain size were obtained by ball milling, obtaining grain sizes between 0.3-0.6 mm, (purity degree of 99.5 %). For smaller particles of quartz the grinding was performed in wet medium containing water and ethanol 1:1 ratio. The ratio material:balls:liquid was 1:1.5:0.4, the speed of rotation was (55 rotation/minutes). The size and shape of the particles were analyzed on a TESLA – 500 electronic microscope.

The grain size distribution was determined at the COULTER-COUNTER machine and the specific areas that correspond to each granulation were determined through the BET method.

Colloidal silica powders. In order to study the influence of the micro reinforcing grains upon the composites' proprieties, we used the imported colloidal silica (Degusso) with the grain size between 0.05 – 0.2 µm and the specific area of 180 m^2/g.

With all these powders we formed a series of liquid-powder composites determining after polymerization the compressive strength and abrasion resistance. The strength tests were performed on cylindrical bars having the diameter of 4 mm and the height of 8 mm, after keeping its for a week at 37^0C in water. We mention that the strength tests were made on 10 bars for each sample.

The powder-liquid composites consists of:

- Monomer mixture: 2.2-bis[4-(2-methacryloyloxypropoxy)-phenyl] propane (Bis-GMA) 65% (obtained in our laboratory) and triethyleneglycol dimethacrylate (DMTEG) 35% (Sigma Aldrich Chemia Grubh, Steinheim – Germany), where the polymeric accelerator was dissolved, N,N-bis (2-hydroxyethyl)-p-toluidine;
- Initiating system made out of: polymerization initiator POB – benzoil peroxide 0.3% de and polymerization accelerator DHEPT – N.N-(2-hydroxyethyl-p-toluidine) 1%;
- Different reinforcing systems: quartz, colloidal silica, quartz + colloidal silica.

The abrasion resistance tests of the composite were executed on bars with the same sizes as for the compressive strength test. The influence of the specific area of the powder on the wear abrasion resistance was studied, by rotating the bars on the surface of the abrasive paper No. 400 at constant pressure and rotating speed. The length of each distance followed by each sample on the surface of new abrasive paper was kept constant for each bar (15 cm). The wear degree was calculated by the loss of volume of the cylindrical bar after erosion taking into consideration the initial and the final sizes (diameter and height) of the bar.

RESULTS AND DISCUSSIONS

Technical characteristics of used quartz and silica in comparison to the ones of the inorganic phase from the enamel and dentine are presented in table 1. From the table 1 one can se that the hardness, tensile and compressive strength, and the elasticity modulus for quartz and silica are higher than the one of the enamel and dentine. The coefficient of thermal expansion has close values for enamel, dentine and colloidal silica, but for quartz is higher. Refractive index is also close enough for all the tested materials.

Grain size distribution is a function of the specific area of the powder. The data for quartz are presented in fig. 1. In what concerned the grain size distribution of the powders with specific area between 1.45 m^2/g and 3.3 m^2/g, one can observe the existence of a wide field, in which the diameter of the particles was between 1µ and 50µ. Between those limits the amount of the powder with smaller particles or larger ones is different from a powder to another. When the specific area increases, the amount of the powder with particles with diameters under 10 µ increases to, while the amount of powder with diameters bigger than 10 µ gets smaller.

Table 1

Technical characteristics of quartz and silica in comparison to the ones of the inorganic phase from the dentine

Material	Enamel	Dentine	Quartz		Colloidal Silica
Chemical composition [% wt]	Ca 7.8 P 17.7 Mg 0.45 CO_2 2.5	Ca 35.5 P 16.7 Mg 1.80 CO_2 3.9	SiO_2 99.56 Al_2O_3 0.08 CaO 0.03 MgO 0.02 Na_2O 0.04 Fe_2O_3 0.025 K_2O 0.01	Si 4 6.53 Al 0.042 Ca 0.021 Mg 0.012 Na 0.033 Fe 0.017 K 0.008	SiO_2 99.999
Density [g/cm^3]	2.9	2.9	2.65		2.24
Knoop Hardness	320 – 343	65 - 70	710 – 790		500
S_t [kgf / cm^2]	103	500 – 525	900		800
S_C [kgf / cm^2]	980 – 3940	2100 – 3500	12300		10000
α 10^{-6}	10 – 11	10 – 11	26.4		11.5
E [kgf / cm^2]	97 – 830	76 – 193	800 – 1400		700
Refractive index n_D	1.60	1.56	1.553		1.691
S_t is tensile strength; S_c is compressive strength; α is the coefficient of thermal expansion; E is elasticity modulus					

The influence of quartz specific area on the appearance of the dental composite material is presented in table 2. Surface of the composite materials get smoother with increasing of quartz specific area (the amount of particles smaller than 5 µm (45.2 % - sample 7 with specific area of 3.2 m^2/g in comparison with 13 % - sample 1 with specific area of 1.45 m_2/g).

Table 2

The influence of quartz specific area on the appearance of the dental composite material

Sample	I	II	III	IV	V	VI	VII
Specific area [m^2/g]	1.45	1.9	2.1	2.2	2.5	2.9	3.2
Surface appearance of the composite	coarse	coarse	slightly smooth	slightly smooth	smooth	smooth	fine

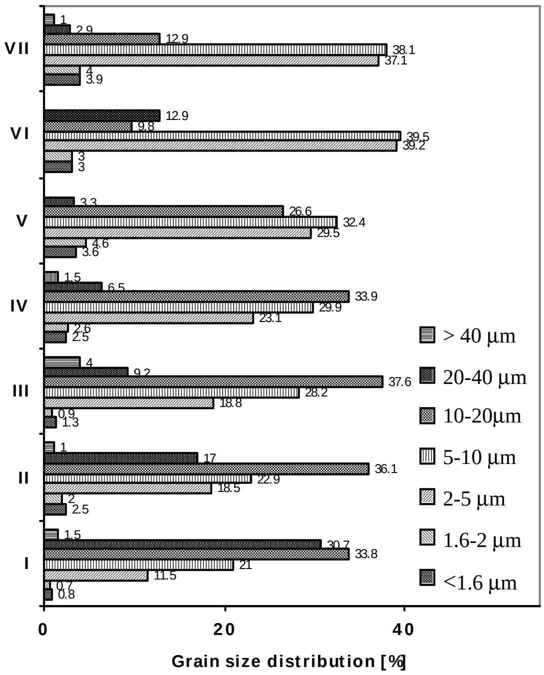

Fig. 1. Grain size distribution as a function of specific area for quartz samples presented in table 2.

In table 3 the specific area, and powder concentration of reinforcing materials for dental use composites is presented.

The compressive strength values for composites reinforced with quartz, colloidal silica and quartz/silica mixture (table 3) is presented in fig. 2.

From fig.2 can be seen that the highest value of the compressive strength was registered in the case of the composite obtained with the powder containing quartz having the specific area 2.1 m^2/g. The value of the compressive strength for the composite with reinforcing material of colloidal silica is below the average level of the one of the composites having quartz as reinforcing material (for charging of 47.3%). Because of the small sizes of the silica particles in the case of this system composite powder-liquid, it was not possible to achieve a good homogeneity of the two phases. By mixing the materials, more and more air is inserted into the composite. Air inclusion leads to smaller values for compressive strength. In the cases of the composites with quartz and silica a decreases of the value of compressive strength can be observed. The highest value can corresponding to the composite with the charging degree of 77%. Remarkable is the fact that at a small variation of the filling's charging degree, do not cause significant difference from a composite to another as we expected. Compressive strength varies between 2358-2536 kgf/cm^2.

Table 3

Specific area and powder concentration of reinforcing materials for dental use composite

Material	Specific area [m^2/g]	Powder [% wt]
Quartz I	2.1	75
Quartz II	2.5	75
Quartz III	2.9	75
colloidal silica	180	47.3
Quartz + 2% silica		77
Quartz + 10% silica		75
Quartz + 20% silica		73.7

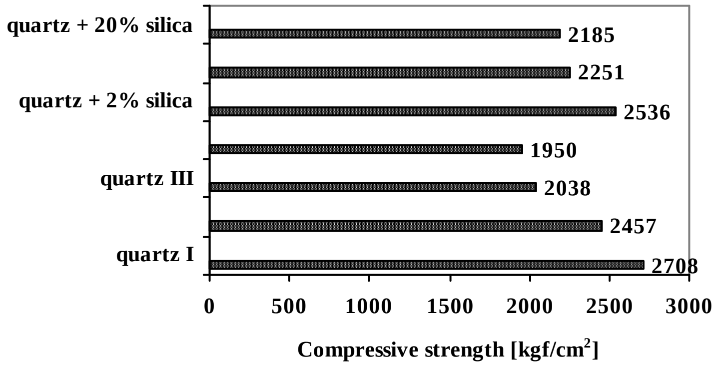

Fig. 2. The compressive strength values for composites reinforced with quart, colloidal silica and quart with silica mixture

The values calculated for the abrasion resistance is given in table 4.

Table 4

Abrasion resistance as a function of specific area for quartz

Material	Specific area [m^2/g]	h_o [mm]	D [mm]	Δh [mm]	Average h loss [mm]	V loss [mm^3]	V loss [%]
Quartz	1.9	8.21 8.30	4.50 4.82	0.66 0.66	0.66	11.251	8
	2.1	8.36 8.22	3.88 3.88	1.02 1.00	1.01	11.936	12
	2.5	8.22 8.40	3.88 3.88	1.24 1.26	1.25	14.772	15
	2.9	8.20 8.22	3.90 3.82	1.50 1.32	1.41	17	16.491
h_o is initial bar height; d is bar diameter; Δh is height loss; V is bar volume							

It can be observed an increasing of the loss of volume as far as the specific area of the quartz powder grows from 1.9 to 2.9 m^2/g. The smallest abrasive wear was observed at the composite which contains as reinforcing material quartz powder with specific area of 1.9 m^2/g.

The electron photographic images presented in fig. 3 a representing the colloidal silica powders, the fluffy appearance can be observed as well as the agglomerates of the particles that couldn't be separated between them. Fig. 3 b showed the irregular shapes of the quartz particles.

CONCLUSIONS

The studied quartz powders take part in the class of the macro fillings, presenting values of the composites' mechanical strength that are between the limits previewed for this type of materials.

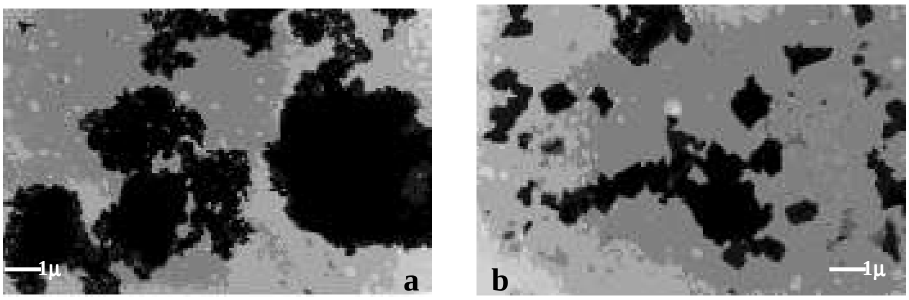

Fig. 3. Micrographs powder: a) Colloidal silica *b*) Quartz powder

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