

Evaluation of a light cured dental composite containing a radiopaque glass filler

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Light curing dental composite pastes were prepared using an indigenously developed Bisphenol A-glycidyl methacrylate (BIS-GMA) resin and a commercially available radiopaque glass filler of different particle sizes. SEM examination of the virgin filler and composite surface revealed agglomeration of particles in virgin state and fine encapsulation of the filler particles in resin matrix of composite. Variation of the filler content shows that an amount of 220 phr (parts per hundred of resin mixture) of the radiopaque glass filler of larger grain size or 250 phr of the smaller grain size incorporated into the paste tends to improve the compressive strength and diametral tensile strength considerably compared to composite containing conventional quartz fillers.

DENTAL composites generally consist of a dimethacrylate organic binder resin such as bisphenol A-glycidyl methacrylate (BIS-GMA) and an inorganic resin filler such as quartz or glass modified with a silane to effect a strong bonding between the resin and the filler incorporated into the resin matrix. The nature and size of the filler play a dominant role in determining the final properties of the composite and its performance in the oral cavity¹. Filler particles are found to have a particle size $< 50 \mu\text{m}$ in conventional composites whereas microfilled composites contain fillers around 400 nm. Glass fillers with barium or strontium incorporated into it to induce radiopacity is widely used in many composites². These radiopaque composites are widely used in posterior applications where stress-bearing materials are required and easy access to the clinician for examination is denied.

The development of the BIS-GMA resin monomer indigenously has been reported earlier³. Both chemical curing and light curing dental composites using this monomer resin and quartz as the filler were developed subsequently and subjected to exhaustive studies⁴⁻⁷. However, the need for radiopacity for monitoring the restored teeth prompted us to use a new radiopaque glass as filler material and the effect of the amount and particle size of this filler upon the properties of the light curing composites developed is reported here.

BIS-GMA resin synthesized in the laboratory and a radiopaque glass filler (GM 27884, M/s. Schott Glasswerke, Landshut, Germany) was used for the study. Three different grain sizes of the same glass were

used [IDENT-NR-642907304 ($0.7 \mu\text{m}$), IDENT-NR-642907604 ($1 \mu\text{m}$), IDENT-NR-642907407 ($1.5 \mu\text{m}$)]. The composition of the glass is reported as SiO_2 55, BaO 25, B_2O_3 10 and Al_2O_3 10. The particle size distribution of the glass powder was determined using a particle size analyser (Galai Cis, Israel). A crosslinker triethyleneglycol dimethacrylate was used to thin down the viscous BIS-GMA to form the resin mixture and a photoinitiator (-) camphorquinone (both from Aldrich Chemical Co., USA) and other stabilizers were incorporated during paste preparation.

The glass filler particle was silanated using a 1% 3-(trimethoxysilyl) propyl methacrylate (97% Aldrich Chemical Co., USA) solution in acetone to ensure strong adhesion between the filler and the BIS-GMA resin. The solvent was allowed to evaporate at 40°C and the coated filler was subsequently heated at 120°C for 60 min and cooled in airtight containers. The silanated filler was added to the resin mixture in different amounts of 205, 220, 250 and 280 phr. They were mixed well in an agate mortar and pestle till a fine dough-like consistency was achieved. Separate pastes were prepared using particles with different grain sizes.

Compressive strength (CS), diametral tensile strength (DTS) and microhardness (MH) were determined for dental composites prepared by light curing the composite pastes. Specimens of size 3 mm diameter and 6 mm height were made for CS measurements by curing the composite pastes in a brass mould kept between two glass plates and held using a C-clamp. The samples were exposed to a visible light source (440–480 nm wavelength, Caulk the Max, UK) for 60 s each on both sides for curing. Subsequently the samples were released from the mould and the inner surfaces of the cured composite were exposed to the light source for a further 20 s to ensure uniform curing within the core of the material. At least ten specimens of each paste were thus cured and stored at 37°C for 24 h before testing.

DTS specimens of size 6 mm diameter and 3 mm height were prepared similarly using a stainless steel mould. Paste packed in the mould between glass slides were exposed to the same light source as above for 60 s each on both sides for curing. The cured specimens were taken out and stored at 37°C in distilled water for 24 h before testing.

Compressive and diametral tensile strength values were evaluated using a universal testing machine (INSTRON Model 1011, UK) using a reported procedure⁸. The crosshead speed of 10 mm/min was maintained for all testings. At least 6 testings were carried out for each sample and mean and standard deviations calculated. For DTS measurements a minimum of 10 specimens were tested. The load at which break occurred was noted and the CS and DTS values were calculated using the equations

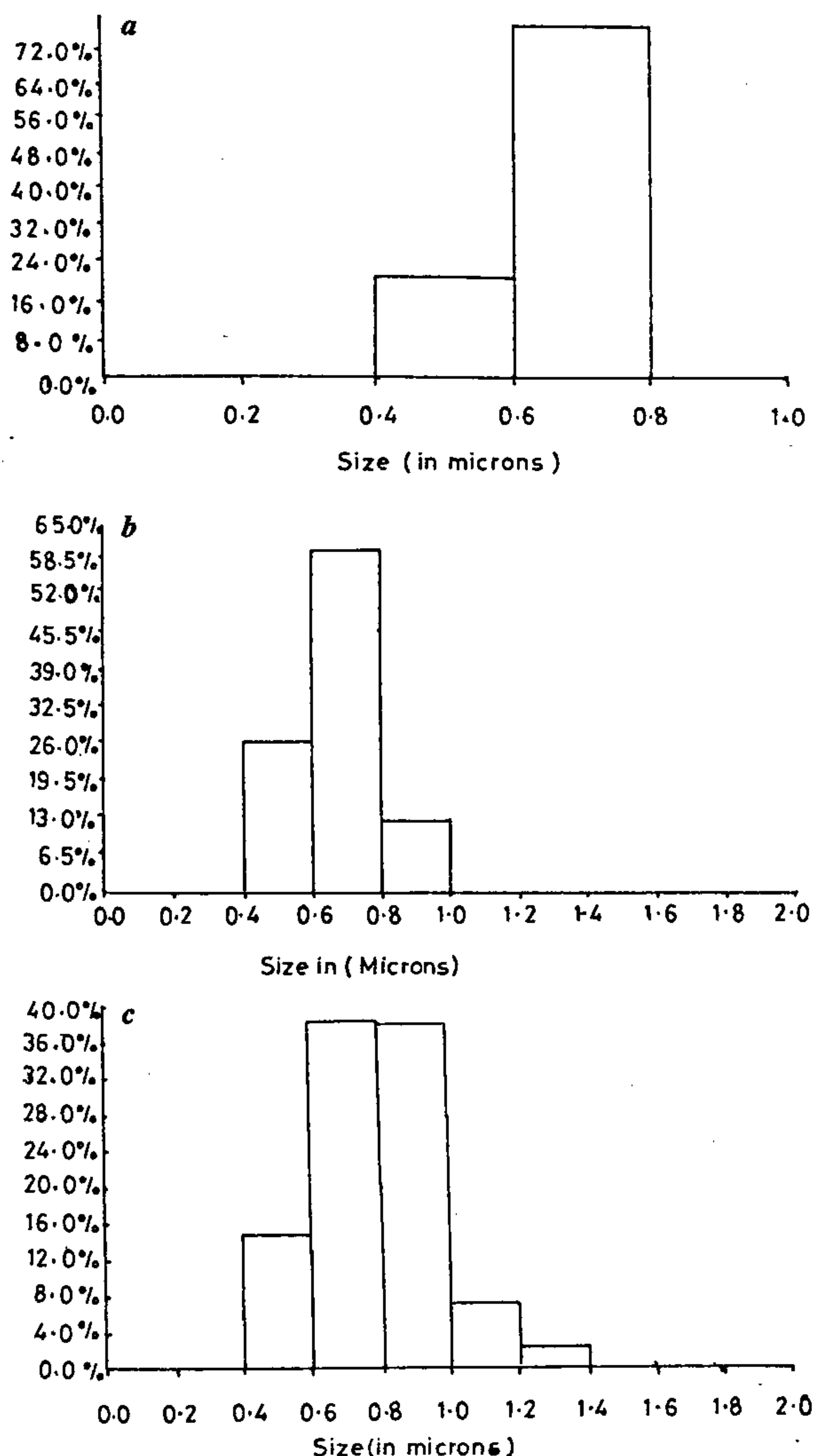


Figure 1. Particle size distribution profiles for glass fillers of grain size 0.7 μm (a), 1.0 μm (b) and 1.5 μm (c).

$$CS \text{ (in MPa)} = P/\pi r^2,$$

where P is the load applied in newtons, r is the radius of the specimen in mm.

$$DTS \text{ (in MPa)} = 2P/\pi DL,$$

where D is the diameter of specimen in mm and L is the height of the specimen in mm.

Specimens for surface microhardness were prepared as was done for DTS using the same mould. For each testing, two specimens were used. The surface microhardness was measured on each side using a Vickers microhardness tester (Carl-Zeiss, Jena). At least 8 meas-

urements for each specimen were done and average taken. The diamond indenter fitted is a square-based pyramid suitable for Vickers hardness measurements. The specimen was placed flat on a glass slide and mounted on a holder on the microscope stage. The specimen surface was examined microscopically and the indenter was then moved into position and the microscope stage was raised steadily until the required load was applied by the indenter upon the specimen. In all cases, a load of 100 g was used. The load was held for 15 s before the microscope stage was steadily lowered. The indenter was then replaced with the objective lens and the image of the indentation was focused. The contrast of the image was optimized using differential filtering and the size of the diagonal of the indentation was measured. The Vickers microhardness (H_v) was calculated using the following equation

$$H_v \text{ (in kg/mm}^2\text{)} = 1854.4 P/d^2,$$

where P is the load applied in gram, d is the length of diagonal in microns.

The surfaces of the composites were polished using silicon carbide papers of 240, 400, 600 and 1200 grit, in that order, and finally with 1 μm alumina powder slurry in water, coated with a layer of gold using a vacuum sputtering machine (E-101, Hitachi ion sputter, Japan) and studied using a scanning electron microscope (Hitachi Model S-2400, Japan, 1500–2000 \times).

Particle size analysis of the three glass powders revealed that the grain sizes ranged from 0.7 μm to 1.5 μm (Figures 1 a–c). It was found easy to incorporate the filler up to a level of 250 phr into the resin mixture. However, incorporating 280 phr of the filler into the resin was found very difficult especially in case of particles with grain size 1.5 μm resulting in pastes of extremely high viscosity. As a result, the mechanical properties could not be evaluated in this case. Similarly it was observed that the paste formed through mixing 205 phr of the filler was of very low viscosity and it was found difficult to cure samples with the light source due to the flowing characteristics of the paste.

Scanning electron micrographs (Figures 2 a,b) show the virgin and composite surface containing glass filler particles. While the virgin filler appears to be agglomerated due to its small size, the filler particles, after silanation, are found encompassed by the resin matrix, which ensures strong bonding at the resin/filler interface. Uniform distribution of filler particles could be observed at the polished composite surface.

Figure 3 shows the compressive strength values for the composite samples prepared using the radiopaque glass filler at different proportions of filler. The recommended values by various standards⁹ and in literature¹⁰ for properties of dental composites are given in Table

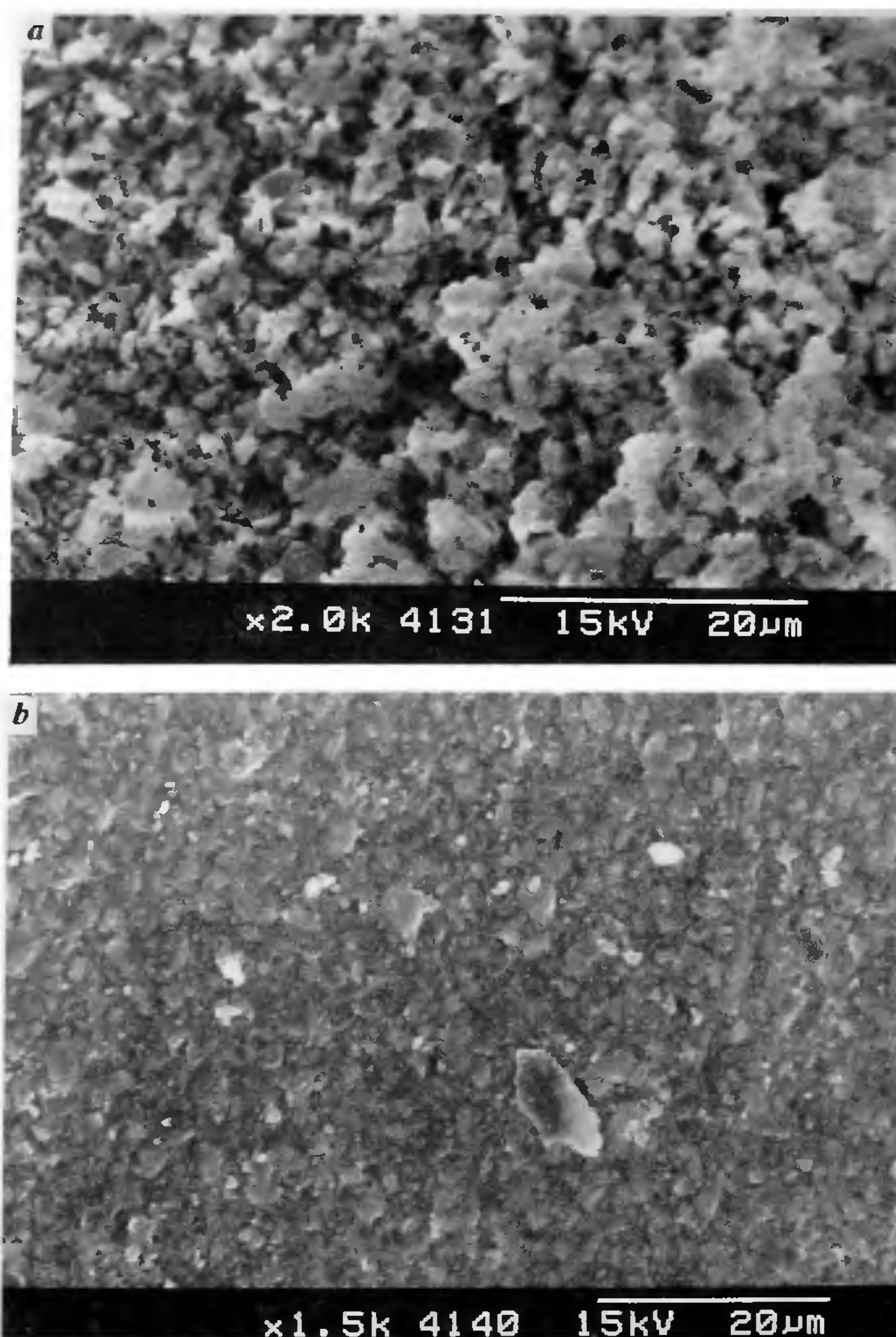


Figure 2. SEM picture of (a) virgin filler in agglomerated condition ($\times 2000$) and (b) dental composite surface ($\times 1500$) showing uniformly distributed fillers encapsulated with the resin matrix.

Table 1. Comparison of values for properties studied in case of light curing dental composites

Property studied	Recommended value ^{9,10}	Values obtained using conventional fillers ³
Compressive strength (MPa)	> 220	220 \pm 6*
Diametral tensile strength (MPa)	> 34	36 \pm 2
Microhardness (kg/mm ²)	> 34	48 \pm 10

*Standard deviation.

1. It can be clearly seen that an amount of 220 phr is found to give excellent CS values in case of particles at all three grain sizes, values reaching up to 300 MPa in certain cases. Higher concentration of filler up to

250 phr though favours composites containing small particles does not seem to agree for particles above 1 μ m grain size as is evidenced in the deterioration of the value.

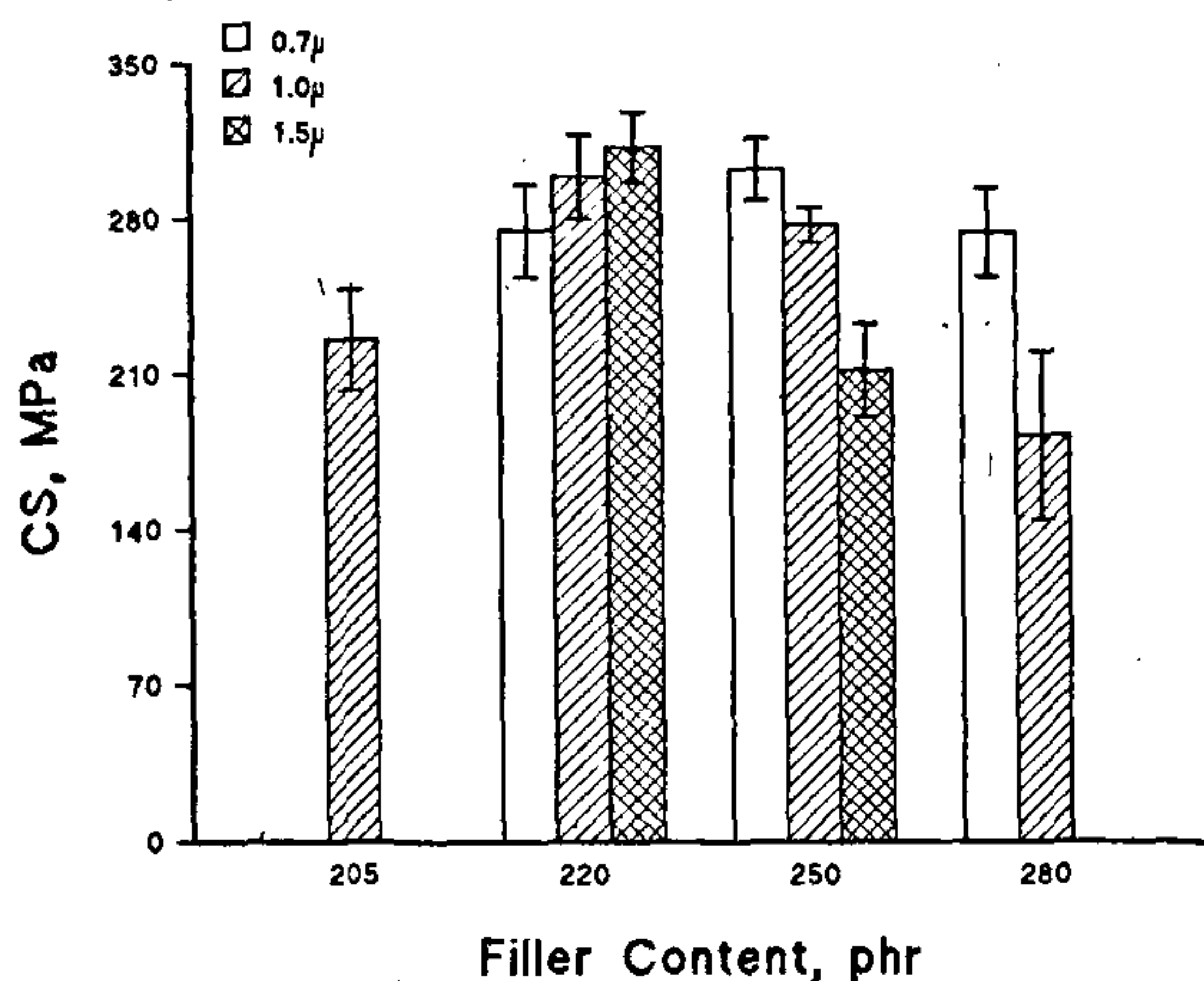


Figure 3. Bar graph showing compressive strength values for composites incorporated with 205, 220, 250 and 280 part per hundred of resin mixture of silanated glass filler GM27884.

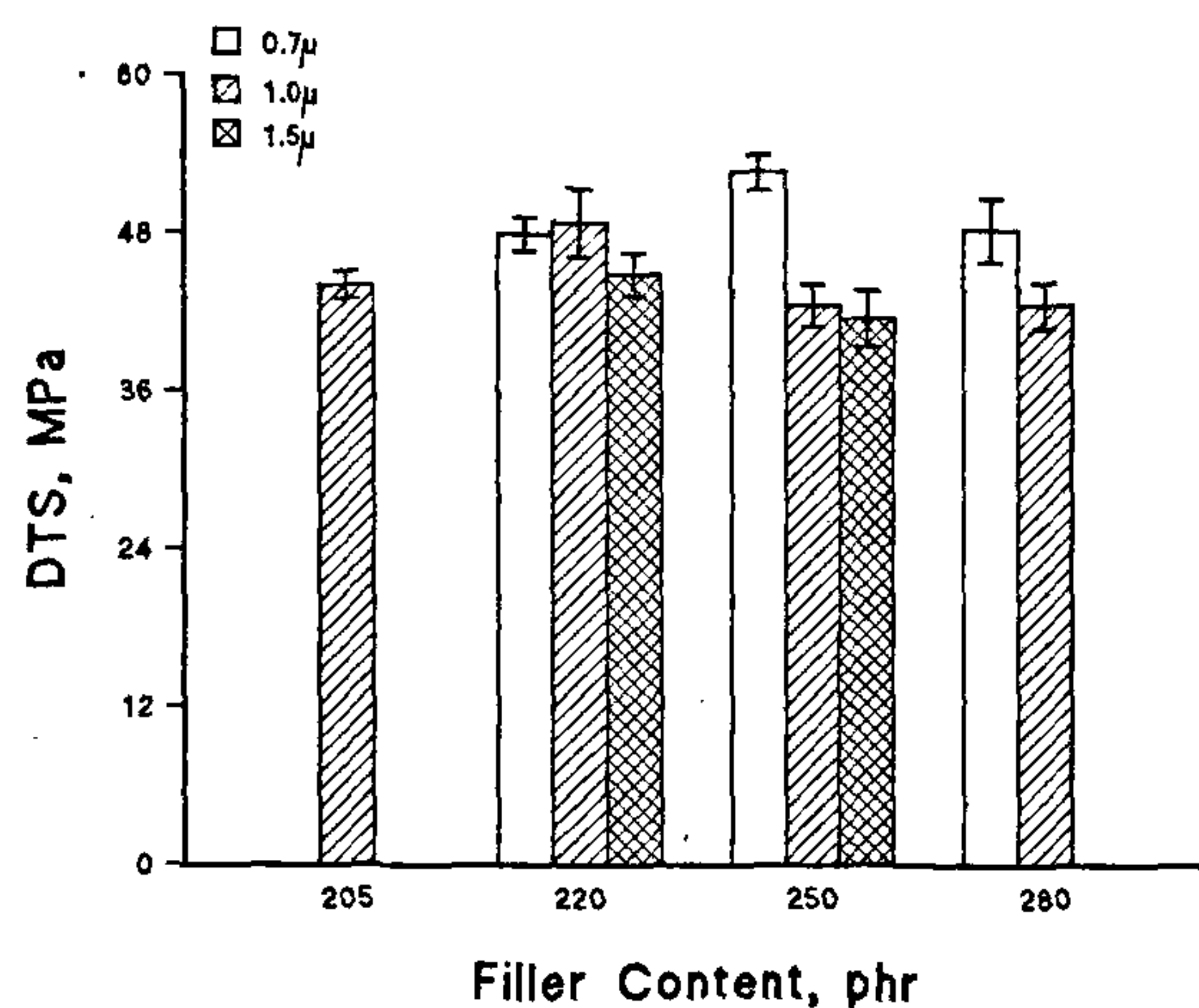


Figure 4. Bar graph showing diametral tensile strength values for composites incorporated with 205, 220, 250 and 280 part per hundred of resin mixture of silanated glass filler GM27884.

Observations of DTS values (Figure 4) also showed the same trend for the composite specimens. Dental composites containing 220 phr of the glass filler tended to show excellent strength values in the range of 45–50 MPa whereas the minimum stipulated value is about 34 MPa. As in the case of compressive strength, DTS was maximum for composites containing 250 phr of 0.7 µm grain size particles though with larger particles, properties tended to deteriorate. Microhardness values also tended to show high values ($> 45 \text{ kg/mm}^2$) at filler content levels of 220–250 phr.

Finally, incorporation of radiopaque glass filler, especially at 220–250 phr range into the composite is found to improve the above properties when compared to the values obtained for composites containing conventional

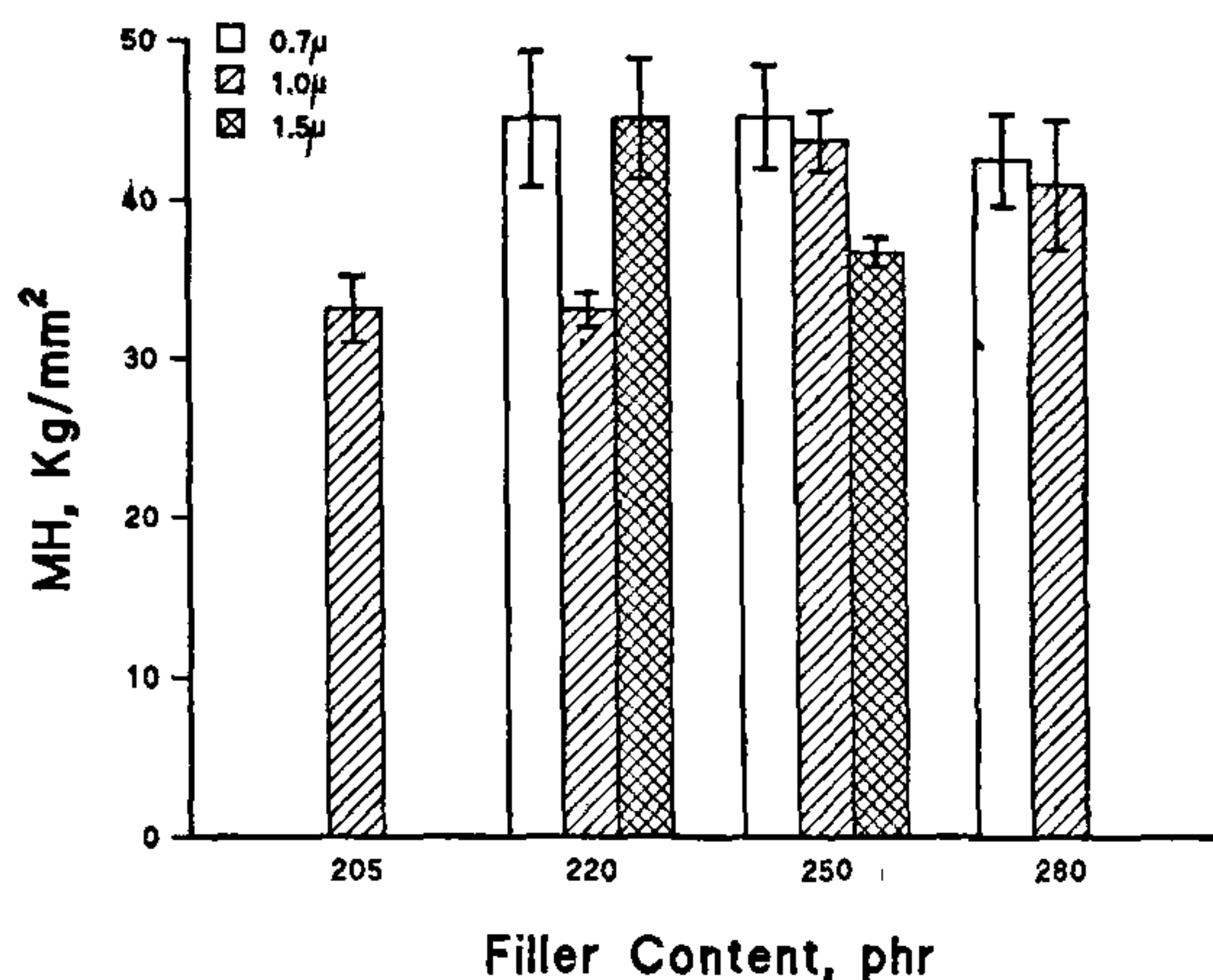


Figure 5. Bar graph showing microhardness values for composites incorporated with 205, 220, 250 and 280 part per hundred of resin mixture of silanated glass filler GM27884.

fillers such as quartz and silica⁵ (Table 1). While compressive strength and tensile strength values are found to appreciate, the microhardness values are found to show comparable results. This can be attributed mainly due to the smaller and uniform particle sizes of the glass filler used (0.7–1.0 µm) compared to the larger and non-uniform size of the conventional quartz fillers (1–50 µm) used in the earlier composite.

In conclusion, about 220–250 phr of the radiopaque glass filler GM27884, varying between grain sizes 0.7 and 1.0 µm, when incorporated into the BIS-GMA resin matrix is found to impart better mechanical properties to a light cured dental composite compared to composites containing conventional quartz fillers.

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