



CHAPTER II

Heat Effect on Light Cured Dental Resin and Mechanical Strength of Conventional Dental Composites

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ABSTRACT

Dental composites are widely used as a restorative material to substitute metallic filling materials such as amalgam. Dental polymer composites are composed of a polymer resin and rigid mineral filler. There are several requirements for the use of filling materials; for example, strong compressive and flexural strength, low shrinkage, good aesthetic appearance, and good resistance to temperature and pH. Dimethacrylate polymer and silica are most commonly used as dental resin and filler for dental composite. Bis-GMA, UDMA and TEGDMA are used as base monomers and a diluent monomer for dental resin preparation. Pure UDMA, mixture of UDMA:TEGDMA and Bis-GMA:TEGDMA 50:50 and 75:25 were mixed with silanized silica at compositions of monomer:fillers 30:70 and 40:60. Heat curing of the monomers were studied by DSC and showed the temperature did not effect on curing from room temperature to 80 °C. Viscosity measurement of monomer showed that increasing in temperature, decreasing in

viscosity. There is no significant different in DTS of dental composites and between the monomer mixture ratio 50:50 and 75:25. The filler content seems to play more roles on the strength than the monomer ratio.

KEYWORDS: Dental Material, Composites, Silanized Silica

1. INTRODUCTION

Plastic filling material or Dental resin composite have become the primary materials used by dentists for replacing the lost hard tissue of tooth. The conventional dental composites or macrofilled dental composites usually consist of glass or reinforced polymer binders (which typically derived from visible light photopolymerization of dimethacrylate monomer), ceramic filler, and adhesive or coupling agent [Phillips and Moore, 1994]. The composition of ceramic filler is usually major to give high load bearing (strength) and varied around 60-80 %wt or 60-70 %volume. This material matches the color of the tooth but have less strength than a metallic filling material such as amalgam. Other disadvantages are high polymerization shrinkage, low resistance to abrasion, and low adhesion to tooth structure. These lead to clinical failure. The filler particle which incorporating in traditional or conventional dental composite has been developed from the macrofiller particle which are mechanically ground or crushed from larger pieces of purely inorganic material such as quartz, glass, borosilicate, or a ceramic (irregular shape and sizes ranging from 1-100 μm) to smaller, softer and more round macrofiller which sizes ranging from 1-5 μm [Bowen, 1962; Leinfelder, 1989; Lutz & Phillips, 1983]. Another types of filler are microfiller and hybrids or blended filler.

Microfiller particles are finely dispersed radiolucent glass spheres chemically created by hydrolysis and precipitation. Originally, the average size of these filler particle was 0.04 μm or 0.05 μm . The colloidal silica or pyrogenic silica ranges between 0.001 and 0.1 μm were used to fill as microfiller in microfiller dental composite. The dispersion is colloidal, therefore, any particle smaller than 0.1 μm are colloiddally dispersed [Bowen, 1962; Lutz & Phillips, 1983]. Hybrid or blend dental composite contains colloidal silica particles in addition to the larger filler particles. Roulet *et al.* (1978) reported that hybrids contain particles with average size of 0.8-1.0 μm . Interestingly nearly all composite resins on the market contain sub micron-sized particles [Leinfelder, 1989]. To induce matrix/filler interaction, the filler is usually impregnated in the surface with a bifunctional coupling agent. In most composites, the filler particles are impregnated with a silane coupling agent [Arcis *et al.*, 2002].

Polymer binders are used for filler embedding and usually epoxy or acrylate types. These particular polymers are good adhesives and can provide crosslinking to enhance strength and good polarity compatible with ceramic fillers. Rigid chemical structure of a monomer often possesses high viscosity such that it is difficult to handle. Practically, more than one type of monomer, e.g. one high viscosity and one low viscosity, is mixed at various ratio to bring about suitable viscosity (easy to use). Effect of monomer type and ratio on polymer conversion (and thus on strength of the composite) using FTIR and near infra-red (NIR) techniques was reported by Stanbury and Dickens (2001ab). On the other hand, Vaidyanathan *et al.* (1992), McCabe (1985) and Imazato (2001) have mentioned that differential scanning calorimetry (DSC) or

differential thermal analysis (DTA) is more convenient to determine polymer conversion (the obtained results are comparable to those by infrared) and study the kinetic. The usable range of conversion found in commercial dental composites is 55-75%. The conversion increases with less content of the high viscosity monomer. Different monomer exhibits different reactivity and the lower viscosity one tends to be more reactive. Photopolymerization kinetics depends on the viscosity of the dental base monomers. From the study of Stanbury and Dickens, Bis-GMA (bisphenol A bis[2-hydroxy-3-methacryloxypropyl]ether) showed the maximum polymerization rate at 10 % conversion. However, UDMA/TEGDMA (urethane dimethacrylate/triethyleneglycol Dimethacrylate or UT) resin reached to higher rate of polymerization at higher degree of conversion than that of the Bis-GMA resin. This is due to lower monomeric glass transition temperature ($T_g = -41.7$ and -6.6 °C for UDMA and Bis-GMA) and lower viscosity of the uncured resin (UT) than those of the Bis-GMA/TEGDMA (BT) resin. Nevertheless, Stanbury and Dickens (2001b) found that resin temperature was raised after light curing. The heat possibly affects on curing condition. Moreover, most work has emphasized on the effect of monomer type and ratio on the conversion but on the final mechanical properties has not been reported. Zhao *et al.* (1997), Johnston *et al.* (1994) found brittle failure for dental composites; however, the fracture toughness of the composites (75-84 %wt mixed filler) was larger than that of resin. The mechanism of the failure is thought to involve viscoelasticity of resin, increased fracture surface area and crack length, and short particle distance inhibiting plastic deformation and elasticity.

In this work, heating effect on curing and viscosity of monomer was discovered. The macrofilled dental composites using silanized silica were characterized. The surface of silanized silica has already treated with silane coupling agent to improve bonding strength between the silica and dental resin. This study focuses on effect of monomer type and ratio (50:50 and 75:25 %wt) and filler content of 60-70 %wt on mixed monomer properties and mechanical properties. The mechanical properties of the prepared conventional composites were compared with the commercial available ones.

2. MATERIALS AND METHOD

The monomers employed in this work were Bisphenol A bis(2-hydroxy-3-methacryloxypropyl)ether (Bis-GMA) and urethanedimethacrylate (UDMA or UD) were supplied by Esstech Co. (Essington, PA) and were used as received. Triethyleneglycol dimethacrylate (TEGDMA), camphorquinone, dimethylaminoethyl methacrylate (DMAEMA) were purchased from Fluka. Switzerland. A commercial silanated Lantan-Alumino-Silicate glass ceramic with high radiopacity (4.4 mm Al-eq.-thick) for dental application, glass powder number GM31684, were obtained from Schott Glass, Germany and used as received. It has high density of 2.89 g/cm^3 , water stability in class 1, low thermal expansion coefficient from 30 to 250 °C of 0 ppm/K, and refractive index 1.579. Commercial dental composites (Ameologen[®] and Aelite fil[®]) were supplied by Nudent Co., Ltd. Thailand. The structure of monomer is shown in Figure 1.

(Figure 1)

2.1 Dental resin preparation

Dental resins were prepared by mixing the base monomer and comonomer in the ratio of 75/25 and 50/50 by %wt using a four-twisted blade turbine on ALC-sL mixer head (variable speed upto 1500 rpm). They were activated for visible light photopolymerization by the addition of 0.2 %wt camphorquinone and 0.8 %wt dimethylaminoethyl methacrylate (initiator weight ratio = 1:4). Prepolymerized dental resin was blended with silanized silica by mixture machine at 500 rpm for 20 minute at room temperature.

2.2 Viscosity measurement

The various ratios of dental resins were poured into sample chamber of Brookfield viscometer model RVDV- III, with a small sample adapter (SSA 21/13 R) and water bath. The measurement was performed from 30°C to 80°C by controlling the temperature in the water bath. The sample was left until reaching the required temperature then the viscosity value was read within 3 minutes.

2.3 Curing temperature measurement

The curing temperature of dental resins with initiators (by heat only) was determined by Perkins Elmer Different Scanning Calorimeter. Samples of 1-3 mg were put in aluminium sample pans and the heating rate used was 1°C /min from 30°C to 90°C with nitrogen gas purge at 20 ml/min.

2.4 Microstructure studies

Filler particles were studied by Cu K- α X-ray diffractometer Rigaku D/MAX-2000 series at 40 kV/30 mA. The standard sample holders were applied to the powder sample compared to commercial silica. The experiment was operated in the 2θ range of 5-70 degree at the scan speed 5 degree/min with 0.02 degree 2θ -stepwise increment. The microstructural analyses were performed by using a JEOL 520 scanning electron microscope (SEM) at a voltage of 10 kV and 500x magnifications. The morphology of the prepared dental composites was investigated also by SEM. Particle size was measured by Mastersizer X ver. 2.15, Malvern with range lens 45 mm. Fourier transform infrared (FTIR) spectroscopy was performed to obtain the chemical structure of silanized silica compared to commercial fumed silica. The samples were prepared by pellet technique and the spectra of these fillers were determined in the wave number range of 4,000-400 cm^{-1} using Bruker Equinox 55/s FTIR spectrometer.

2.4 Diametral tensile strength

The general tensile strength test method (ASTM D638) is not suitable for measuring the tensile strength of brittle materials. An alternative method, the diametral tensile strength (DTS) test was therefore utilized. DTS test specimens were prepared in a cylindrical stainless steel mold, then loaded to failure with a crosshead speed of 10 mm/min, using 25 kN load cell. DTS were determined by the following standard formula (1)

$$DTS = 2f/(\pi dl) \dots\dots\dots(1)$$

Where f = load at failure; d = specimen diameter; l = specimen thickness

The specimens were prepared by pouring the unpolymerized dental resin into a stainless steel mold. Resin tablets (3 mm thick by 6 mm diameter) were photopolymerized between glass slide and translucent polycarbonate strip in a dental curing unit (3M Curing Light XL3000) with a light intensity of approximately 300 mW/cm² at approximately 470 nm measured by a radiometer for 40 seconds of each side. They were subjected to test in a Universal Testing Machine following the procedure in American Dental Association-Specification No. 27 for Direct Filling Resins. Five specimens for each sample type were tested and their mean values were determined.

2.5 Microhardness test

The microhardness of the sample was measured using FM-700e Digital Microhardness tester (Future-tech Corp. Japan) with 500 gram load, dwell time 15 sec and 136° pyramid diamond indenter (Vicker Hardness scale). The specimens were prepared in the same way as the DTS specimen followed by polishing the surface with 0.3 μm slurry alpha alumina (Imptech, South Africa). The pyramidal shaped indenter applied the surface of specimens within dwell time then the force was removed. In the Vickers method, the indentation length of vertical and horizontal axis is measured and average. The experiment was repeated five times for each sample set of parameters. The impression length was measured microscopically and the test load was used to calculate a hardness value.

3. RESULTS AND DISCUSSION

The results in Table 1 show that when temperature increases, the viscosity of all dental resin systems decreases.

(Table 1)

The viscosity of UDMA resin is much less than that of Bis-GMA resin and when adding diluent monomer TEGDMA, this mixture becomes less viscous. It is obvious that the viscosity of Bis-GMA/TEGDMA is more than the viscosity of UDMA/TEGDMA at the same monomer ratio. In general, fillers show poor dispersion in the viscous materials [Shernoy, 1999]. The higher initial viscosity of the Bis-GMA base resin makes itself difficult to incorporate the filler into the resin (not good homogeneity). When its viscosity is reduced by addition of TEGDMA or by increasing temperature, more filler content incorporated into dental resin can be achieved such that mechanical properties should be enhanced. Moreover, due to lower viscosity, the filler is incorporated into the UDMA-based mixture more rapidly than in Bis-GMA-based mixture. Furthermore, the polymerization reaction controlled by diffusion or chain flexibility mechanism can occur easier to get higher conversion and conversion rate [Stansbury and Dickens, 2001ab; Lovell *et al.*, 1999a; Lovell *et al.*, 1999b; Silikas and Watts, 1999]. Consequently, more strength (due to high conversion or higher crosslinking) of the polymer phase can contribute to better mechanical properties.

For this experiment, camphorquinone is the curing agent that needs light at wavelength about 470 nm to complete the polymerization. Light curing can be determined by exothermic heat at room temperature using DSC [McCabe, 1985].

Maximum rate of curing is affected by irradiation power. Higher power leads to higher conversion but the relationship is not linear. However, exposure to light can generate heat that may have some effect on curing since heating (light induced thermal rise to 65 °C) was found by Stanbury and Dickens (2001b). Moreover, heating may occur due to viscous shear during mixing. Thus, constant heating program from room temperature to 200 °C was applied to the samples. The DSC thermogram of five dental resins (Figure 2) has shown no significant peak that the resin had turned from liquid to solid or gel when increasing temperature.

(Figure 2)

This confirms that the dental resin cannot be thermally cured, at least over this temperature range. Thus the heat generated by exposure to light can only affect to reduce viscosity [Silikas and Watts, 1999] which affects to increase monomer mobility (away from glass transition temperature of the polymerizing materials) such that the conversion is enhanced [McCabe, 1985]. Besides, this information is benefit to ensure that the preparation of dental composite (before light cure) can also be done at elevated temperature (with reduced monomer viscosity) such that better filler uptaking (or better dispersion) is gained without premature curing. In addition, since the base monomers are highly viscous, the viscous heat dissipation will not cause premature curing upon mixing.

Structure and Morphology of Silanized Silica

Silanized silica is silica that has treated with silane coupling agent to induce matrix/filler interaction. That means higher energy is needed to debond filler particle and resin matrix and such that high diametral tensile strength should be obtained. The morphology observed by SEM is shown in Figure 3.

(Figure 3)

The particles are irregular shape having the mean size of 4.23 μm measured by particle size analyzer which is in accordance with the size seen in the SEM result. Chemical structure of the silanized silica is revealed by FTIR in Figure 4. The hydrophilic peak of hydroxy group typically found on silica surface Figure 4a is diminished and more complex peaks in the region of 1000-1100 cm^{-1} (Figure 4b) corresponding to silane groups or Si-O-R bonds, are more prominent.

(Figure 4ab)

This indicated that there is hydrophobic group present in the silica and the hydrophilic surface is modified such that the silanized silica has better hydrophobic property. Figure 5 shows x-ray diffraction patterns of commercial fumed and precipitated silica in comparison with silanized silica.

(Figure 5abc)

This reveals that the commercial silica (without surface modification) are amorphous glass while the highly radiopaque silanized silica has crystal structure with the strongest peak at $2\theta = 25.66$ degrees corresponding to silica composition. These structures should

provide high hydrophobic surface area for resin bonding with filler and bring about improved mechanical strength due to crystal structure.

Diametral Tensile Strength (DTS)

Although degree of conversion was not reported here, the conversion of our samples is expected to be in the workable range. Lovell *et al.* (1999) reported that using 3.0 mW/cm² for 200 seconds, the conversion of 50/50 Bis-GMA/TEGDMA was 0.60. Stanbury and Dickens (2001b) found that 70:30 mass ratio of BisGMA:TEGDMA gave conversion of 0.689 by NIR and 0.692 by Mid IR at seventh day after exposure to light cure for 60 seconds at irradiation power 65 mW/cm². The conversion of 70:30 %wt Bis-GMA:TEGDMA at 3 min after light curing is 0.673 by NIR. If the monomer ratio changes to 50:50, more conversion to 0.739 and 0.772, at 3 min and seventh day after light curing respectively, was determined by NIR (or 0.743 by Mid IR at seventh day). We use high irradiation power light source (300 mW/cm²) so shorter cure time of 40 seconds could be appropriate to reach the same level of conversion.

Factors affecting the DTS such as monomer ratio, monomer type, and filler content were studied. Sample abbreviations are, for example, BT7246, UT5537, and so on. Base monomer and diluent monomer (denoted by two letters) ratios of 75:25 and 50:50 %wt are denoted by the first two numbers; i.e. 72 and 55 while monomer:filler weight ratio were denoted by the last two numbers; i.e. 37 = 30:70 and 46 = 40:60. Although the viscosity results showed that Bis-GMA could give poorer properties due to high viscosity limiting conversion as mentioned earlier and filler uptaking,

Figure 6 revealed that both composites from Bis-GMA and UDMA monomers have comparable strength.

(Figure 6)

This can be explained by the earlier work reported by Stanbury and Dickens (2001a) that although Bis-GMA was less reactive than UDMA while TEGDMA was the most reactive monomer, the final overall conversion (180 seconds for light exposure) of Bis-GMA:TEGDMA (mass ratio 0.64:0.36) was lower than UDMA:TEGDMA (0.62:0.38) about 5%. Moreover, the local conversion (in gel) showed similar degree of crosslinks in both monomers systems despite the more rigidity in chemical structure of Bis-GMA causing sluggish motion. This small difference in conversion may not effectively alter strength. This also reveals that the rigidity of Bis-GMA structure can compete with higher conversion and better filler uptaking of UDMA. Their strengths are rather low compared to those of commercial ones. This is possibly a consequence of more complex components in the commercial ones, e.g. with colloidal silica or mixed filler, than our samples that contain only macrofiller [Zhao *et al.*, 1997].

Additionally, the previous work by Chowdhury *et al.* (1995) reporting that most of dental restorative materials contained mixtures of Bis-GMA and TEGDMA at 60:40 %wt to yield a high degree of polymerization while Shajii and Santerre (1999) reported the composition for optimal conversion was 75:25 %wt. However, for these systems monomer ratio has slight effect on DTS. The diametral tensile strength from monomer ratios 50/50 and 75/25 are not significantly different although the earlier work stated that more diluent content caused more conversion and thus more strength is expected. On the

other hand, the difference in degree of overall conversion due to monomer type and ratio has less significance on strength because of its low content (minor phase). Strength of the composite possibly relates to filler content, a major phase that functions to bear load mainly.

Both composites from different monomers show the same trend that filler content plays more important roles in determining the strength than monomer type and ratio. It is obvious that at the same ratio of monomer and comonomer, increasing the filler content reduces the tensile strength. From the previous work for composites with both macrofiller and microfiller by Htang *et al.* (1995); Zhao *et al.* (1997); Johnston *et al.* (1994), higher filler loading (loading about 60-84 %wt) in dental composites can lead to high mechanical properties (e.g. DTS, fracture toughness, flexural modulus) determined by conventional mechanical test. Unlike those works, i.e. instead of having the composite of the highest filler-resin ratio (70/30) being the strongest composite due to high content of filler, the composite having 60 %wt filler content shows better strength. This was proved to be true for the pure UDMA resin composites (uniform monomer reactivity) in Figure 7 although the viscosity of UDMA is higher than those of mixed monomers.

(Figure 7)

In general, the enhancement of diametral tensile strength can be achieved if the particles are well bonded to the resin matrix. Polymer matrix can spread on silica via condensation reaction between silanol group and hydrolyzed silane molecules [Lim *et al.*, 2002]. The unexpected results can be explained by poor filler distribution and more

filler agglomeration. According to SEM results of the cryo-crack surfaces for UD37 and UD46 in Figure 8a-d,

(Figure 8a-d)

the above explanation cannot get along because the micrographs reveal rather uniform filler distribution in both system with more filler density in UD37 than in UD46 due to higher filler content. The holes represent the location of filler particles which were detached out leaving the smooth surface holes (resin-filler interfaces). This implies that silanized silica sufficiently provide hydrophobicity for good filler dispersion but the adhesion of matrix and the silica is not so strong as evident by adhesive or brittle failure in the hole [Johnston *et al.*, 1994]. The most prominent evidence for the failure mechanism is shown in Figure 8cd. It is the different mode of failure; one (UD37) shows brittle failure and another (UD46) shows plastic deformation or ductile failure that consumes more crack energy contributing to higher strength. The brittle failure associates with short particle distance causing high strain rate in this region since the particles are rigid and assumed to be non-deformable under this stretching condition. Since the particle distance is short and fast extension, the deformation of the resin occur in the elastic region but crack is simultaneously initiated in the resin-filler interface then propagates into resin region such that the crack occurs earlier at low tensile force (brittle failure). The ductile failure exhibits plastic deformation on the crack surface. This can happen due to longer inter-particle distance causing lower strain rate; upon stretching, resin is extended beyond its yield point allowing plastic deformation and crack is simultaneously initiated at interface and propagates into the resin. So this transition from

ductile to brittle failure limiting by inter-particle distance confines the optimum filler loading for optimum load bearing capacity or strength.

Microhardness of Dental Composites

Hardness of the conventional composites is important as an indicator for wear or erosion resistance. It was studied as a function of filler content, monomer type and ratio. Mean value and standard deviations of Vicker hardness for the conventional composites are presented in Figure 9.

(Figure 9)

Hardness values of BT composites are higher than those of UT composites. This is clearly due to more molecular rigidity of Bis-GMA than that of UDMA. The monomer ratio shows some effect on improving microhardness while the filler content exhibits better control on surface rigidity. In other words, Figure 10 clearly shows increasing TEGDMA reduces the microhardness for 70 %wt filler and increases microhardness for 60 %wt filler. To distinguish the effect of filler, the composites of pure UDMA and silanized silica at 60-70 %wt were tested and the results are shown in Figure 10.

(Figure 10)

It clearly sees that the resin is much softer than the composites and the composite hardness increases with filler content or decreases with monomer content. Microhardness of conventional composites is also lower than that of pure UDMA-based composites and the commercial dental composites. This is probably due to the effect of the flexibility of diluent monomer TEGDMA rather than low conversion because

viscosity of pure UDMA is much higher than BT72 and BT55. The large hardness value difference between filler loading 60 and 70%wt of BT72 compared with BT55 due to the ten times higher viscosity of BT72 than BT55 so that the poorer mixing and more inhomogeneity can be obtained.

4. CONCLUSION

The strength and hardness of the conventional composites are mainly dependent on filler content rather than monomer type and monomer ratio, which can be manipulated to gain suitable viscosity. The optimum ratio between filler and polymer resin can provide ductile failure that result in maximum diametral strength. The higher filler content in resin brings about surface hardness. That means the dental composite with higher content of filler provide the aesthetic but not endure for the mastication force.

5. ACKNOWLEDGEMENTS

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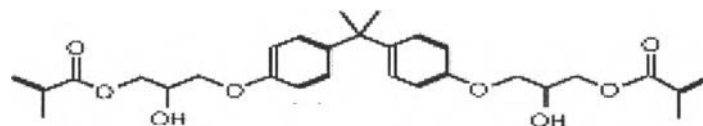
Table 1. Viscosity properties of prepolymerized dental resin.

Sample	Viscosity (cps) at Temperature ($^{\circ}\text{C}$)					
	30	40	50	60	70	80
UDMA	2944	1663	844	375	181	87.5
UT75/25	294	178	106	56.3	31.3	15.6
UT50/50	40.6	25	15.6	9.38	3.13	0
BT75/25	1678	800	416	209	119	65.5
BT50/50	116	62.5	40.6	25	12.5	6.25

^aMeasure at the same speed = 80 rpm. Shear rate = 27 sec^{-1}

^bEssetech, Certification of product analysis. UDMA viscosity = 7850 cps and Bis-GMA = 1,976,000 cps. (Brookfield Instrument)

Base monomer

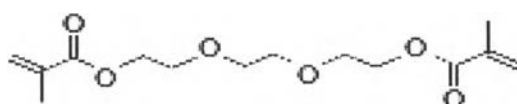


Bis-GMA



UDMA

Diluent monomer



TEGDMA

Figure 1

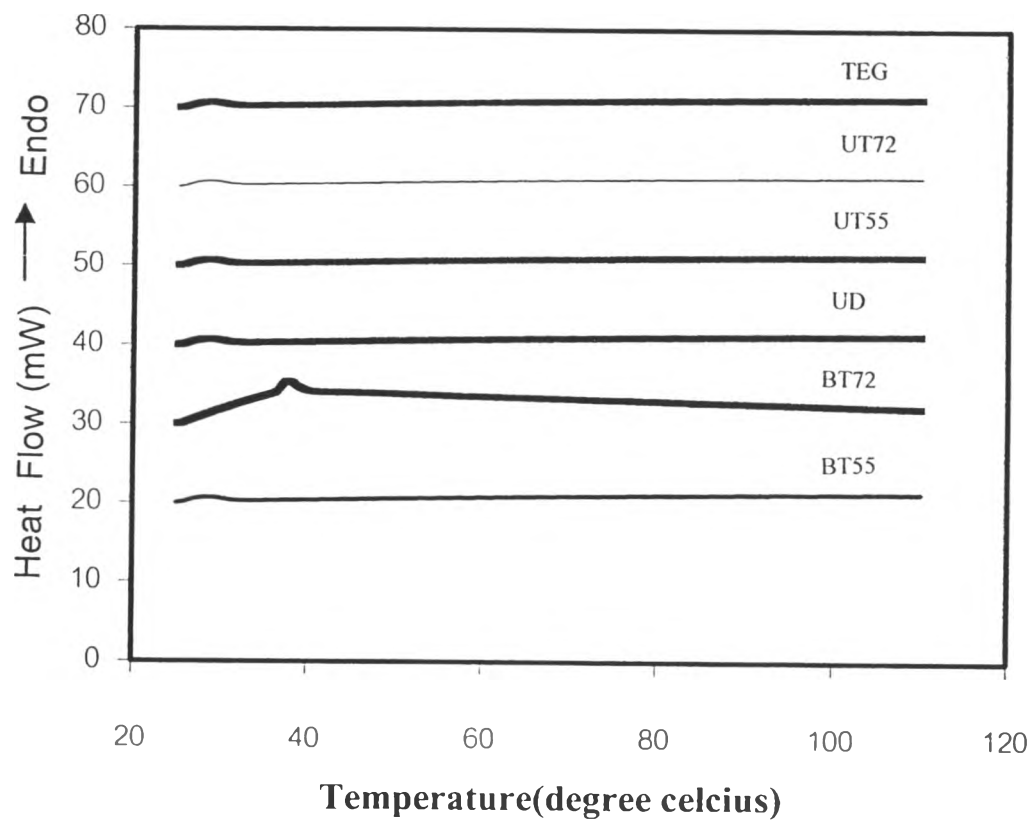


Figure 2

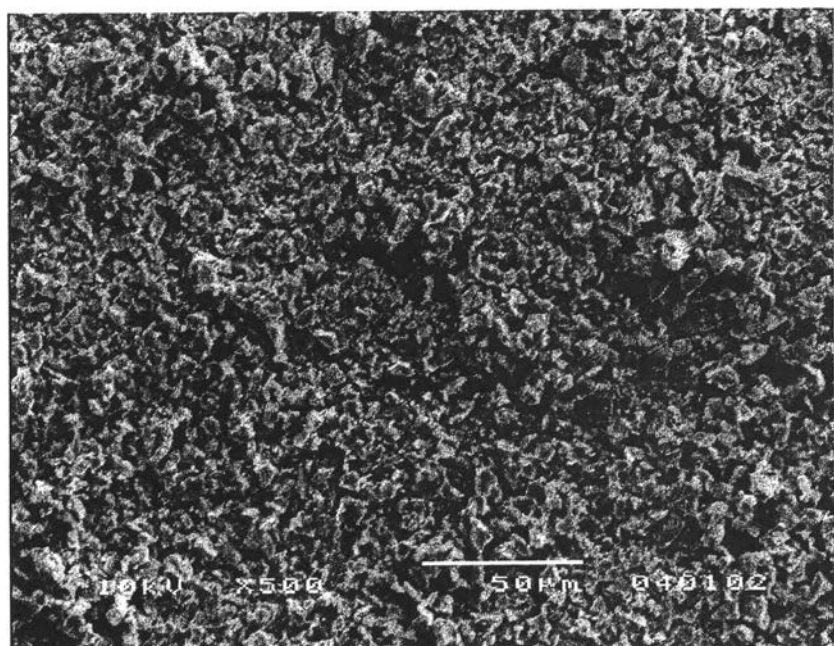


Figure 3

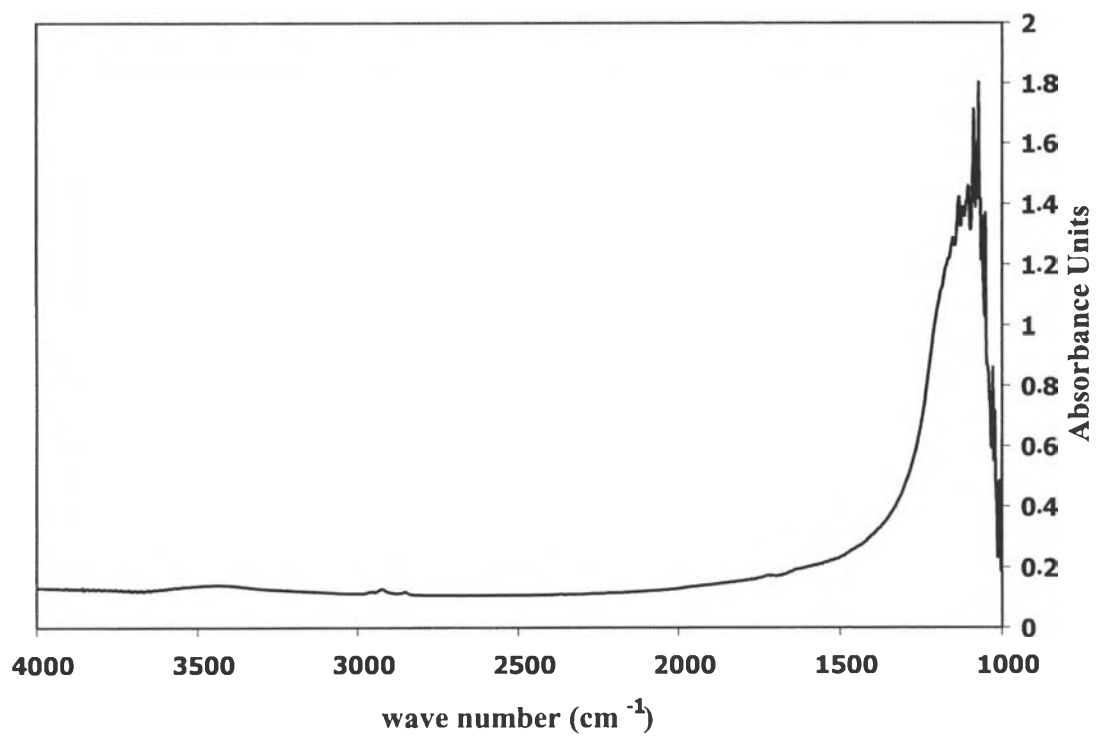


Figure 4a

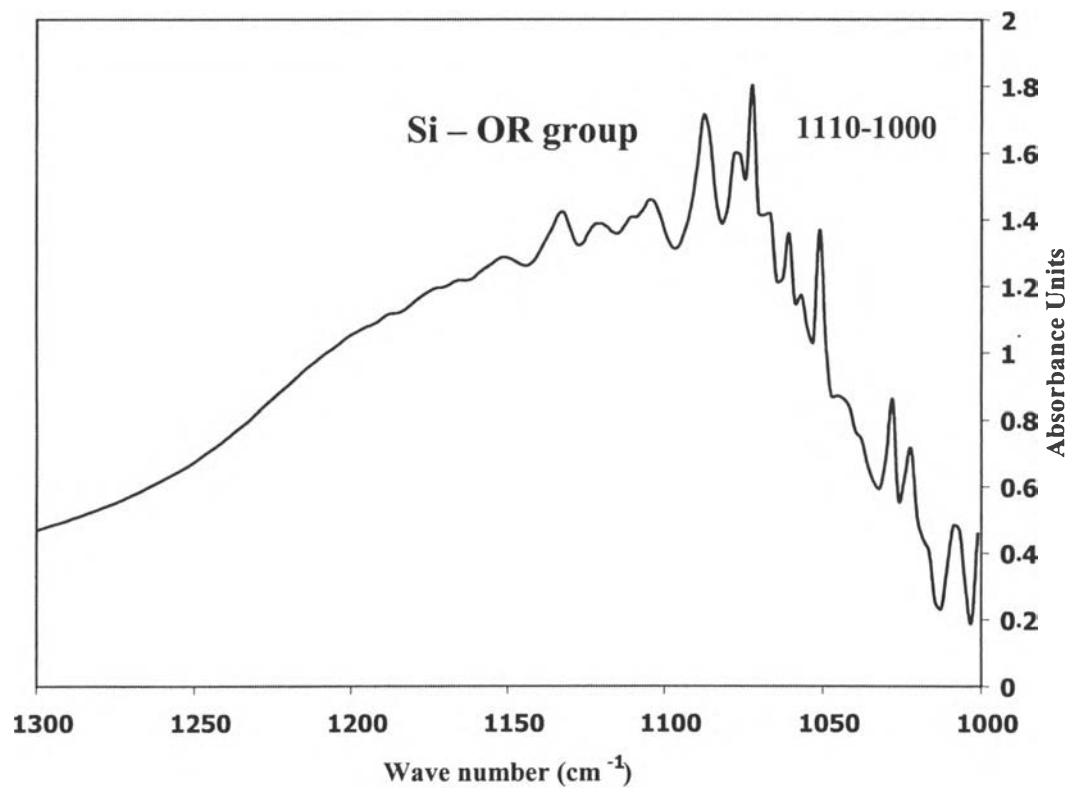


Figure 4b

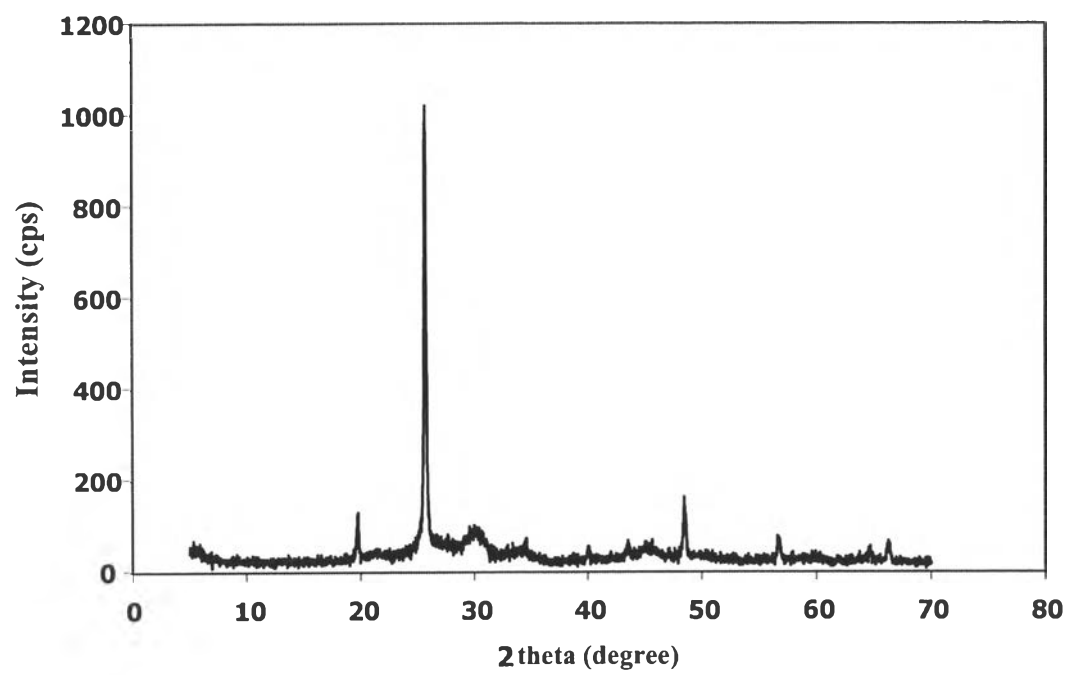


Figure 5a

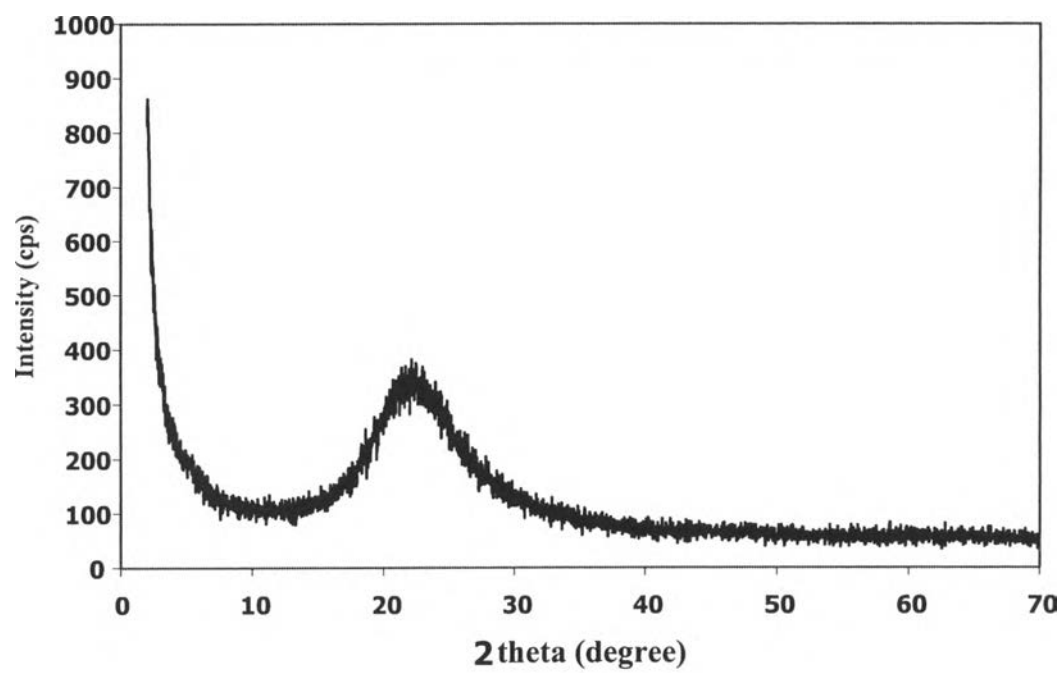


Figure 5b

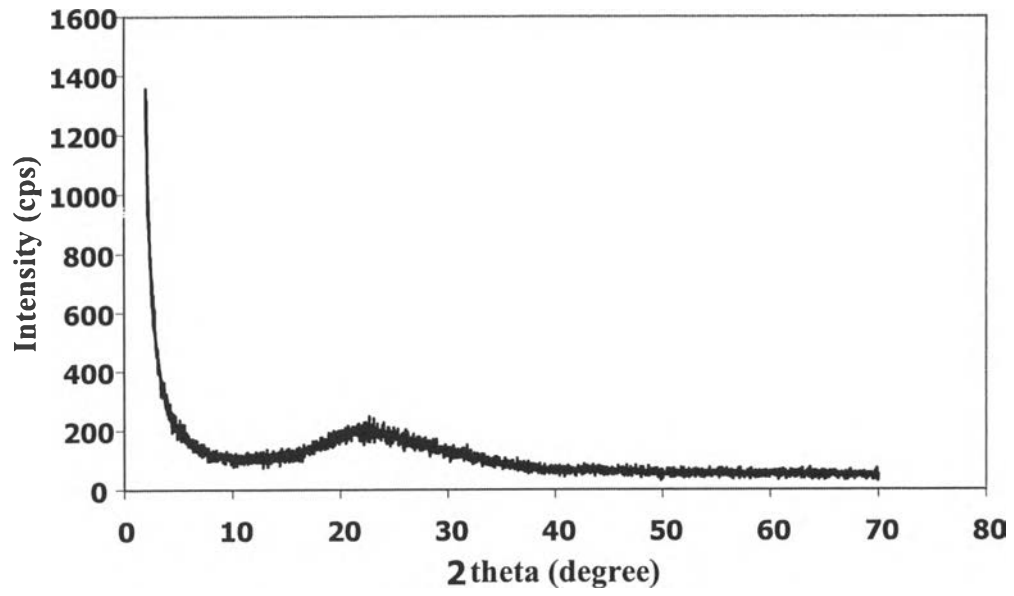


Figure 5c

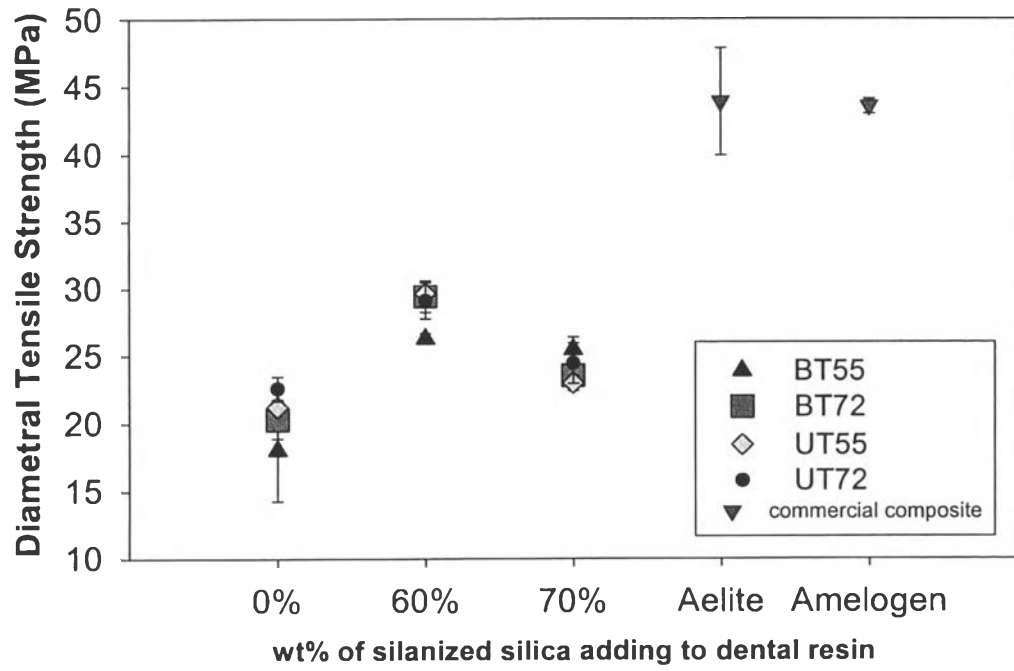
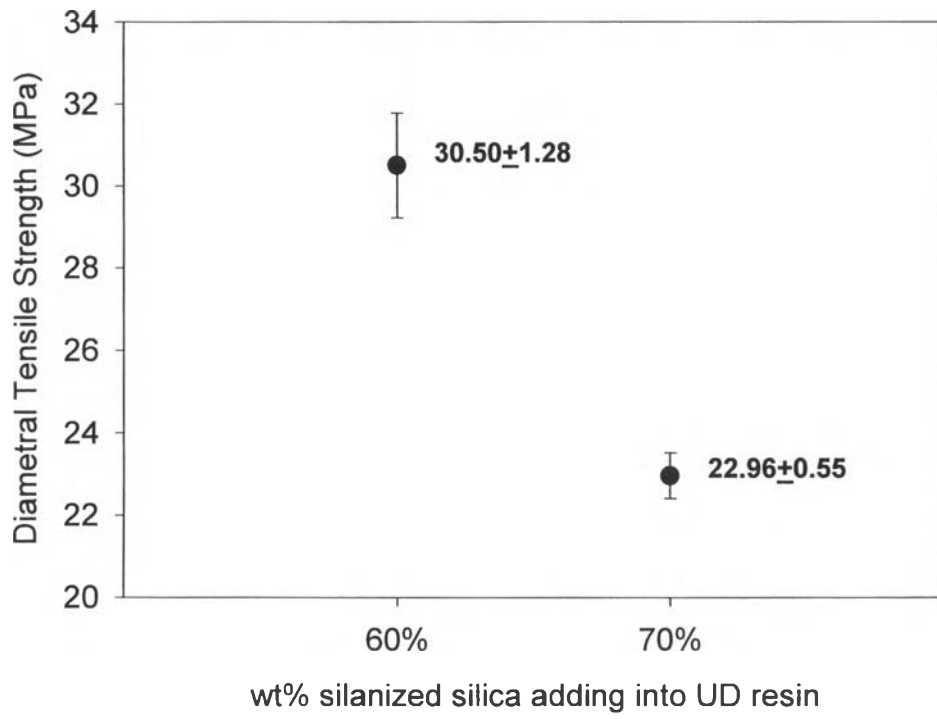
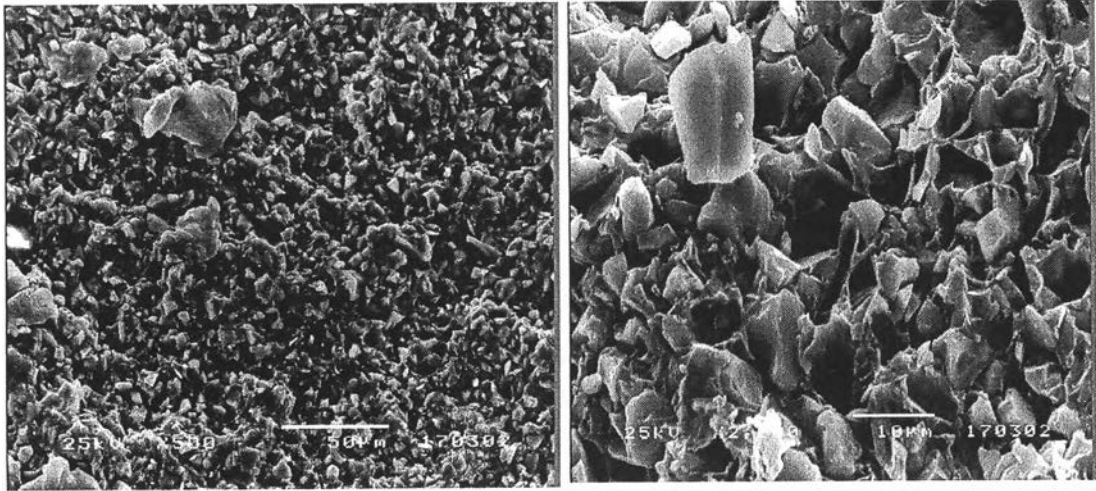


Figure 6

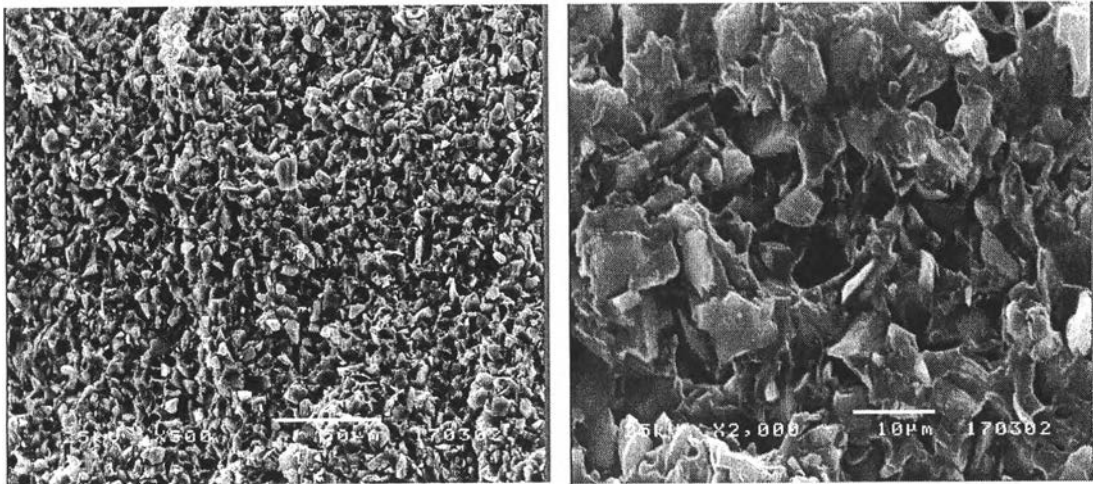


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a.

b.



c.

d.

Figure 8 a-d.

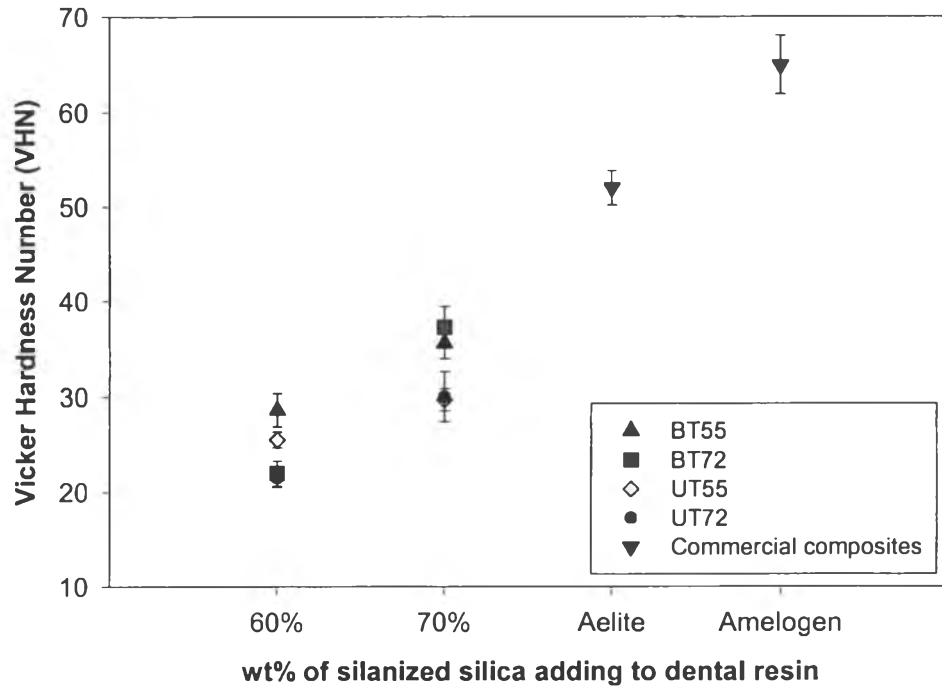


Figure 9

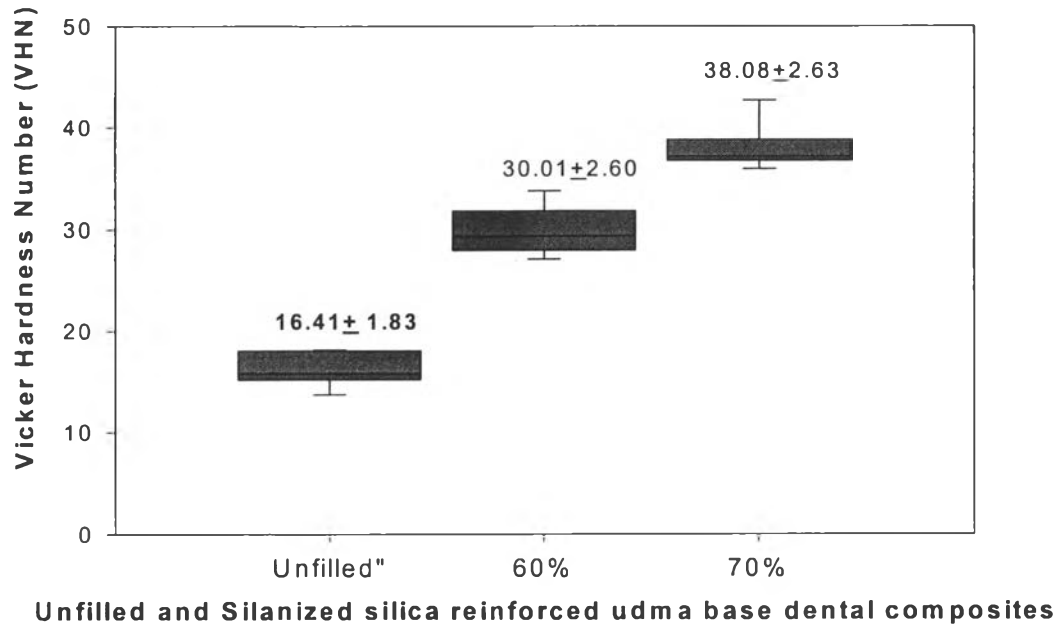


Figure 10