

# Testing Different Properties of A Light-Cured Denture Base Material After Addition of Silicon Oxide Nanofiller (An in Vitro Study)

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## ABSTRACT

**Background:** Improving the properties of heat-cured and self-cured acrylic resin have been studied by many researchers. However, little studies concerned with visible light cured resin (VLCR) improved through addition of nanofiller are available. The purpose of this study was to evaluate some properties of (VLCR) after addition of SiO<sub>2</sub> nanofiller.

**Materials and Methods:** SiO<sub>2</sub> nanofiller were added to (VLCR) tray material after being dissolved in tetrahydrofuran (THF) solvent. According to the pilot study 2% SiO<sub>2</sub> nanofiller addition exhibited better properties than the other percentages (1%, 3%). The main study conducted involved (100) specimens divided into 5 groups according to the test included. (20) Specimens were selected for each test (10 samples for the control group and 10 samples for the experimental 2% SiO<sub>2</sub> nanofiller group). The properties investigated were transverse strength, impact strength, surface hardness, surface roughness, water sorption and solubility. Scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) were used to assess nanofiller distribution and identification of elements. The data were subjected to descriptive statistical analysis and independent sample t-test.

**Results:** The mean value of transverse strength of experimental group increased significantly, while the impact strength of experimental group decreased significantly when compared to control group. A significant increase in surface hardness was noticed in the experimental group, while non-significant increase in surface roughness was observed. The water sorption values were decreased significantly, while a non-significant decrease in water solubility was observed in the experimental group.

**Conclusion:** Addition of SiO<sub>2</sub> nanofiller to (VLCR), slightly improve the transverse strength and surface hardness, while water sorption and solubility slightly decreased. The impact strength was significantly deteriorated, while the surface roughness shows non-significant increment.

**Key Words:** Visible light cured resin, SiO<sub>2</sub> nanofiller, nanocomposite. (J Bagh Coll Dentistry 2017; 29(1):47-54)

## INTRODUCTION

Generally poly (methylmethacrylate) (PMMA), either heat cured or self-cured is the basic material in complete or partial denture removable prosthesis. Little amount of residual monomer always remains relying upon curing technique<sup>(1)</sup>. A residual monomer content about 1.81-1.85% in heat polymerized PMMA has been reported by Hiromori<sup>(2)</sup>. On the other hand, a higher amount of residual monomer left in self-cured PMMA, about 2-6% has been reported. Despite the fact that PMMA indicate low solubility in water, remaining monomer may spread into the oral mucosa<sup>(1,3)</sup>.

The little amount of solubility is a consequence of non-polymerized monomer and water-dissolvable added ingredients. Considerable number of patients with hypersensitivity reaction to PMMA have been reported<sup>(3-5)</sup>.

Visible light cured resins (VLCR), based on urethane dimethacrylate emerge as one of the optional material in removable prosthesis rather than the conventional PMMA because of their synthetic nature are devoted from monomethacrylates<sup>(4,6)</sup>.

Light polymerization technique do not need complicated laboratorian methods and the possibility of hypersensitivity reaction is much reduced because methylmethacrylate monomer is not present in (VLCR)<sup>(7-11)</sup>.

One of the pioneer in manufacturing of VLCR was known as Triad based on urethane dimethacrylate (UDMA). It was introduced because of simple laboratory procedure, decreased bacterial colonization, biocompatible material, patients preference, possibility to bond to another resin and obviously do not need mixing and proportioning techniques<sup>(10,11)</sup>. Nevertheless, its practical uses was restricted due to inherent brittleness and reduced impact strength<sup>(12)</sup>. An advanced generation of methylmethacrylate free monomer was manufactured by Densply Trubyte.N.Y, known as (Eclipse). This light polymerized product comprises 3 types of materials; denture base plate resin, Teeth arrangement resin and teeth contouring resin. The first one will become the final denture base after light curing, the second will be utilized for setting of teeth and will be bonded to the denture base by light curing. Finally, the contouring resin will be adapted on the denture base resin, arrangement resin and cervical portion of artificial teeth, afterward will be polymerized by light<sup>(6)</sup>.

AL-Mulla et al<sup>(12)</sup> conduct a study concerning some physical and mechanical properties of

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VLCR. They found that Triad (VLCR) was harder and more rigid than the conventional heat and self-cured PMMA, but the impact and transverse strength exhibit less values. Ali et al found that the surface hardness, flexural strength and flexural modulus of UDMA (Eclipse) light and heat cured resins were more significant than (Meliodent) heat cured and self-cured (Probase cold) denture base materials<sup>(6)</sup>.

AL- Taie and khamas found that the transverse strength of repaired VLCR were inferior when compared to Ivomat pressured cold cured resin and conventional heat cured resin<sup>(13)</sup>. Akin et al concluded that VLCR possess comparable cytotoxicity to the conventional PMMA, nevertheless VLCR shows less water sorption when kept in water for long time<sup>(14)</sup>.

In the modern biomaterial, nanotechnology has gained a considerable importance due to their characteristics properties and structures. The nano materials technology shown to be valid in 1980, " indicating to zero-dimension, one-dimension, two-dimension, three dimension materials with a size less than 100nm". There are usually four types of nano materials; nanofiber, nanopowder, nanoblock and nanomembrane. The nanopowder research is more extensive and its production is more advanced<sup>(15,16)</sup>.

The high surface energy, huge surface area, minute size and a large magnitude of surface atoms are the main characteristics features of nano materials. Moreover, nano materials possess four unique and superb effects " quantum size effect, quantum tunneling effect, surface effect and minute size effect" <sup>(17)</sup>. The addition of nano particles such as silica, clay, calcium carbonate , zirconium oxide to polymers are of interest for the researcher because of remarkable changes in physical properties <sup>(18-22)</sup>.

Silicone oxide nanoparticles ( $\text{SiO}_2$  ), otherwise called nano silica or silica nanoparticles are highly recommended in biomaterial research because of their low cost, stability, low toxicity, and the capability to be grafted with a lot of polymers <sup>(23,24)</sup>. Micro or nanofiller addition can significantly improve the absorption of energy and associated properties of hybrid or composite materials. Nanofiller like  $\text{CaSiO}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{SiO}_2$  ,  $\text{ZrO}_2$ , Clay nano particles, single walled or multi walled carbon nanotubes are of interest in nano composite engineering <sup>(24)</sup>. Although the improvement in the properties of VLCR are in progress, yet limited studies in the literature are available concerning the mechanical or physical performance of VLCR through addition of nanofiller. A study conducted by Qasim et al <sup>(25)</sup>, reported a significant increase in the impact and

flexural strength of VLCR reinforced with carbon nanotubes. Specimen's discoloration as the percentages increase was the main drawback of this study. The goal of the present study is to examine different properties of VLCR such as transverse strength, impact strength, surface hardness, surface roughness, water sorption and solubility after addition of  $\text{SiO}_2$  nanofiller.

## MATERIALS AND METHODS

A pilot study was conducted to estimate the proper amount of nanofiller to be added to VLCR. Silicon dioxide ( $\text{SiO}_2$ ) nanofiller coated with epoxy (American element Co. USA) was added to tray VLCR (Hoffmann's Germany). At first 37gm of VLCR was dissolved in 25ml of Tetrahydrofuran (THF) solvent (Biosolve-chemicals, the Netherlands) in 250ml glass beaker placed on magnetic stirrer(BOECO, Germany) with hot plate for 60 min <sup>(25)</sup>. A digital electronic balance (KERN, Germany) was used to weigh the material before mixing. Then, the  $\text{SiO}_2$  nanofiller were added in (1%, 2%, 3%) by weight respectively, while 0% represents control group. After mixing was completed, the mixture was loaded into heat resistant plastic mold specially made for this study to make a sheet of 4mm thickness and preserved in laboratory hood away from light for one day. Then the mold transferred to a dissector for 2 days and after that dried in vacuum oven ((HYSC, Korea) at 60 °C for 3 hours to complete evaporation and dryness of the material. Later on, the sheet was removed, kneaded well with fingers and cut into small pieces to be adapted into specially made transparent plastic mold which has 4 holes, one on each corner. Two types of mold were made, the first one has 3 rectangular cavities (80mmx10mmx4mm); length, width and thickness respectively, prepared by CNC machine (Computer numerical control) for impact strength. The second mold has also 3 rectangular cavities (65mmx10mmx2.5mm), for transverse strength, surface hardness and roughness. After coating the mold with Vaseline, the lower transparent cover was also coated with Vaseline and reassembled with the middle part, then the material was adapted carefully in the mold and excess material removed by sharp knife; then the upper cover placed on the mold and tightened with screw and nuts so that the middle part is sandwiched between the two transparent covers. The mold was transferred into the light cure chamber(KT-888, China , Wavelength of 400.um) so that the upper surface left for 7.5 min, then the mold was inverted and cured again for another 7.5 min(total curing time 15min). When curing was completed

the pattern were removed carefully, then finished and polished except samples of surface roughness was not polished.

The samples of water sorption and solubility was prepared by cutting metal discs with dimensions of (50mm and 0.5mm), diameter and thickness respectively. Two discs were placed on transparent plastic plate after being coated with Vaseline. After that a stone was poured around the discs and on the borders with vibration, then a second transparent plate was seated upon the stone, thus the discs and stone were sandwiched between two transparent plastic plates. The two plates were tightened gently with screw and nuts. After setting of the stone, the screws were opened and the upper plastic plate removed, then the metal discs removed carefully and the resulted mold cavity surrounded by stone was coated with separating medium. After that, a little sheet of VLCR was adapted into the stone mold, then the second plastic plate was applied and tightened with screw and nuts and transferred into curing chamber and the same procedure was repeated as mention previously.

According to the pilot study in which 24 samples were made, 12 for the transverse strength and the other 12 for surface hardness. Three samples were made for each percentage (0%, 1%, 2%, 3%). The results shows that the 2% nanofiller has greater values than the other percentages, so the decision was to carry on the main study with 2% SiO<sub>2</sub> nanofiller.

#### Main study specimens grouping:

(100) specimens were prepared for this study. The specimens were divided into (5) groups according to the test utilized in the present study. Twenty samples were selected for each test (10 samples for the control and the other 10 for the 2% SiO<sub>2</sub> nanofiller.

#### Testing procedure:

##### 1. Transverse strength test:

The specimens fabricated were with dimensions of (65mmx10mmx2.5mm), length, width and thickness respectively. After conditioning in water at 37°C for 48 hrs. <sup>(26)</sup>, the samples were subjected to a 3 point bending test using an Instron machine(Laryee CO., Ltd. China). The stress were recorded and determination of the transverse strength follows this formula:

Transverse strength=  $3PI/2bd^2$  <sup>(27)</sup>, where

P: is the peak load

I: is the span length

b: is the sample width

d: is the sample thickness.

##### 2. Impact strength:

Specimens were kept in distilled water for 48 hrs. at 37°C before being tested. Rectangular samples of dimension (80mmx10mmx4mm), were fabricated. The test was performed using Izod impact testing machine (Time testing machine, China) <sup>(28)</sup>. The energy absorbed by the un-notched specimens was calculated using this equation:

Impact strength=  $(E/bd) \times 10^3$  <sup>(27)</sup>, where

E: is the impact energy absorbed in joules.

b: is the width of the sample.

d: is the thickness of the sample

The surface of a fractured portion of randomly selected sample of the experimental group was sputter coated with gold, and tested with a scanning electron microscope (SEM, AIS2300C, USA).

##### 3. Surface hardness:

The samples were prepared with dimension of (65mmx10mmx2.5mm), and stored in distilled water for 48hrs. at 37°C <sup>(26)</sup>. Shore D hardness tester (Elcometer, Germany) with a calibrated scale from (0-100 units) was used. The final value of this test was obtained by calculating the average value of 5 readings performed for each sample. All measurements were done by one person.

##### 4. Surface roughness:

The surface roughness of (VLCR) samples were measured by Profilometer device (TR200, Time CO. China). The sample utilized in this test were prepared with dimension of (65mmx10mmx2.5mm). Later on, the samples kept in distilled water for 48hrs. at 37°C, then the test was performed by Profilometer apparatus that capture surface irregularities. Three readings for each sample were determined and the average value of these readings represents the final value.

##### 5. Water sorption and solubility:

The VLCR samples for water sorption and solubility were fabricated as discs having a dimension (50mm ± 1mm and 0.5mm ± 0.1mm), diameter and thickness respectively <sup>(26)</sup>. A dissector containing dried silica gel was utilized to dry the samples that were kept in an incubator at 37°C ± 2°C for 24hrs. After that the samples were transferred from incubator and kept at room temperature for 1h. , then weighed within accuracy of (0.0001g) using digital electronic balance. The same procedure continued daily in order to reach a constant mass "conditioned mass" (M1), which indicates that the loss in weight from each sample was not exceeding 0.2mg in 24hrs. Subsequently, immersion of the specimens in distilled water at 37°C ± 2°C remained for one

week. After that, each sample was taken out from water with tweezers and blotted with clean towel for 30 seconds, remained in air for 15 seconds and weighed again, the obtained value represents M2. In order to measure the amount of water solubility, the samples were dried again in the desiccator at  $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and the same procedure continues as mention before in sorption test, and the obtained value represents the reconditioned mass M3. Water sorption and solubility were determined by the following formula:

$W_{sp} = (M2 - M1) / S$  ( $W_{sp}$ =water sorption,  $M1$ = conditioned mass,  $M2$ = Mass of specimen after 7 days immersion in water)

$W_{sl} = (M1 - M3) / S$  ( $W_{sl}$ =water solubility,  $M1$ = conditioned mass,  $M3$ = reconditioned mass,  $S$ = surface area of the disc) (26).

The values of the control and experimental groups were subjected to descriptive statistics and inferential statistics using Independent t test with probability level at ( $p < 0.05$ ). The (SPSS) statistical package for social sciences, V 19 was used to perform the results.

## RESULTS

### FTIR, SEM, EDS examinations:

Figure.1, shows FTIR spectrum of  $\text{SiO}_2$ . The Si-o-Si peak is observed at  $1066\text{ cm}^{-1}$ . Figure.2-3, shows the FTIR spectral results of VLCR before and after addition of nano  $\text{SiO}_2$ . The appearance of new peak at  $1049\text{ cm}^{-1}$  due to Si-0-Si. The (SEM) examination of the fractured impact portion are shown in Figure 4-5. Fair homogeneity of nano silica, spherical like matrix and some pores were detected. The presence of  $\text{SiO}_2$  component in the experimental sample was emphasized by (EDS).Si, C, O peaks were revealed on the surface of the sample as shown in Figure 6.

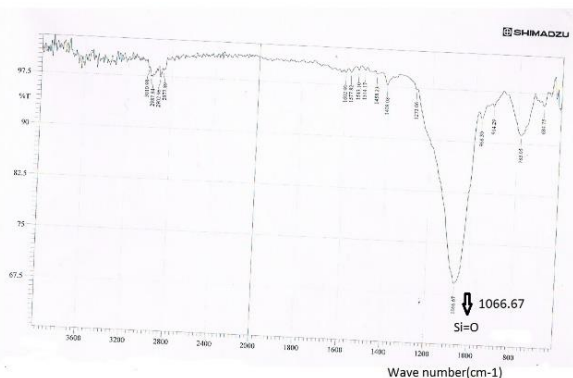


Figure 1: FTIR spectrum of  $\text{SiO}_2$  nanofiller.

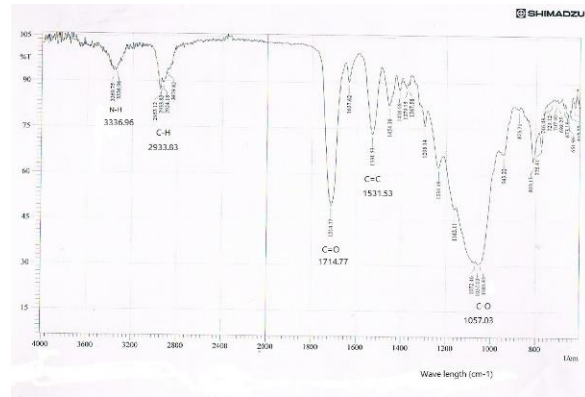


Figure 2: FTIR spectrum of VLCR.

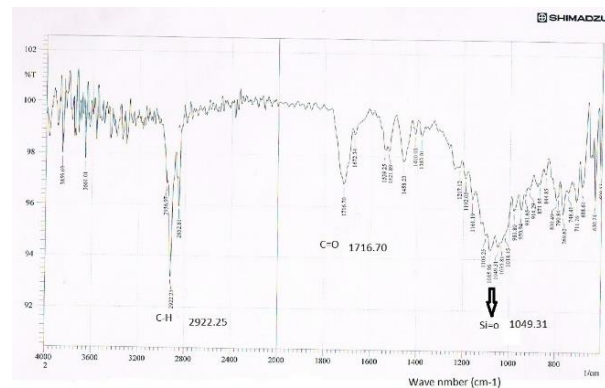


Figure 3: FTIR spectrum of VLCR/  $\text{SiO}_2$  nanofiller.

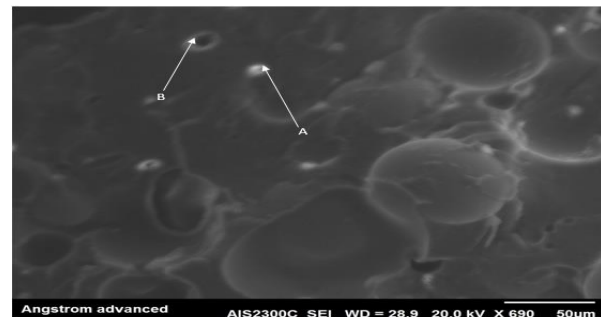


Figure 4: SEM of the fractured impact surface reinforced by 2%  $\text{SiO}_2$  (50um). A,  $\text{SiO}_2$  nanofiller. B, pore.

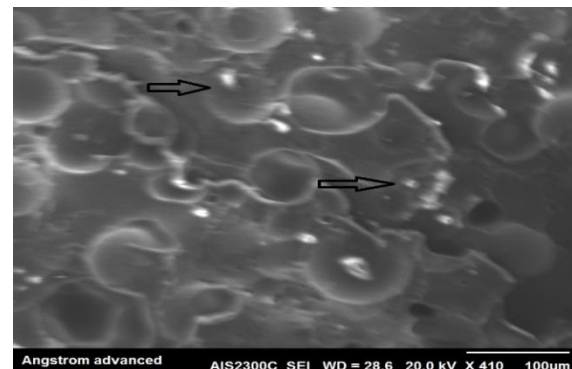


Figure 5: SEM of the fractured impact surface re-inforced by 2%  $\text{SiO}_2$  (100um). Arrows show nanofiller.

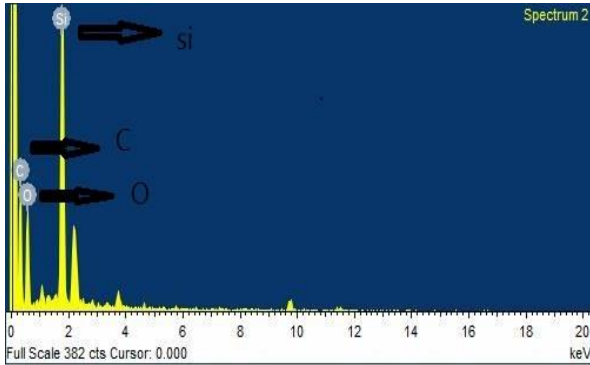


Figure 6: EDS spectra showing Si, C, O elements on the surface of reinforced sample by SiO<sub>2</sub> nanofiller.

**Transverse strength, impact strength, surface hardness, surface roughness, water sorption and solubility:**

The means, standard deviation, minimum, maximum values of the above parameters are summarized in Tables 1-6.

The transverse strength of the experimental group was higher than the control group. T-test revealed a significant difference between the two groups (P< 0.05) as displayed in table 1.

The impact strength of the experimental group reduced significantly when compared to controlled group as listed in table 2. For surface hardness, the experimental group recorded higher mean value than the controlled group. This increment was significant (p<0.05) as shown in table 3.

Table 4. , shows a non-significant increase in surface roughness of the experimental group when compared to controlled group (p>0.05).

A significant decrease in water sorption was observed when 2% SiO<sub>2</sub> nanofiller added to VLCR as shown in table 5. The incorporation of 2% nano silica resulted in a non-significant decrease in water solubility as presented in table 6.

**Table 1: Descriptive summary and T-test for transverse strength (N/mm<sup>2</sup>)**

Groups	Descriptive statistics						T-test for equality of means (d.f.=18)		
	N	Mean	S.D.	S.E.	Min.	Max.	Mean difference	t-test	P-value
Control 0%	10	64.58	4.815	1.521	57.60	74			
Experimental 2% SiO <sub>2</sub>	10	69.53	4.715	1.491	58.3	74.4			

S: Significant at P< 0.05

**Table 2: Descriptive summary and T-test for impact strength (Kj/m<sup>2</sup>)**

Groups	Descriptive statistics						T-test for equality of means (d.f.=18)		
	N	Mean	S.D.	S.E.	Min.	Max.	Mean difference	t-test	P-value
Control 0%	10	6.05	0.743	0.235	5.25	7.5			
Experimental 2% SiO <sub>2</sub>	10	5.25	0.552	0.174	4.25	6.25			

S: Significant at P< 0.05

**Table 3: Descriptive summary and T-test for surface hardness**

Groups	Descriptive statistics						T-test for equality of means (d.f.=18)		
	N	Mean	S.D.	S.E.	Min.	Max.	Mean difference	t-test	P-value
Control 0%	10	84.38	1.70	0.539	81.8	87			
Experimental 2% SiO <sub>2</sub>	10	86.08	1.692	0.535	84	89.6			

S: Significant at P< 0.05

**Table 4: Descriptive summary and T-test for surface roughness**

Groups	Descriptive statistics						T-test for equality of means (d.f.=18)		
	N	Mean	S.D.	S.E.	Min.	Max.	Mean difference	t-test	P-value
Control 0%	10	3.3637	0.602	0.19	2.55	4.25			
Experimental 2% SiO <sub>2</sub>	10	3.4432	0.597	0.189	2.58	4.28			

NS: Not significant at P> 0.05

**Table 5: Descriptive summary and T-test for water sorption (mg/cm<sup>2</sup>)**

Groups	Descriptive statistics						T-test for equality of means (d.f.=18)		
	N	Mean	S.D.	S.E.	Min.	Max.	Mean difference	t-test	P-value
Control 0%	10	0.600	0.0890	0.0281	0.460	0.730			
Experimental 2% SiO <sub>2</sub>	10	0.4949	0.0884	0.0279	0.370	0.630			

S: Significant at P< 0.05

**Table 6: Descriptive summary and T-test for water solubility (mg/cm<sup>2</sup>)**

Groups	Descriptive statistics						T-test for equality of means (d.f.=18)		
	N	Mean	S.D.	S.E.	Min.	Max.	Mean difference	t-test	P-value
Control 0%	10	0.0281	0.0110	0.0035	0.010	0.050			
Experimental 2% SiO <sub>2</sub>	10	0.0184	0.0108	0.0034	0.010	0.040			

NS: Not significant at P> 0.05

## DISCUSSION

VLCR are accepted for many uses in restorative dentistry. However this material exhibit weak mechanical properties, especially flexural and impact strength. An attempt was made to improve some properties of this material through addition of SiO<sub>2</sub> nanofiller because previous studies mention positive changes in the physical and mechanical properties of substrates when nanofiller were added<sup>(18, 19)</sup>.

The resistance to breakage and stiffness can be measured by transverse strength. Flexural (transverse) strength was selected in this study because it resemble the masticatory or loading forces in the oral cavity. The results of the present study shows a slight improvement in transverse strength of VLCR reinforced with 2% SiO<sub>2</sub>. This might be due to nanofiller aids in the continuity of the resin matrix by filling interstitial spaces and dispersion into the polymer matrix. Therefore, the shear strength between nanofiller and (UDMA) polymer chain will be enhanced<sup>(20, 24)</sup>. Another possible explanation since the modulus of elasticity of SiO<sub>2</sub> nanofiller is high, greater resistance to the applied load is expected without deformation leads to increment in flexural strength<sup>(29)</sup>. Moreover, SiO<sub>2</sub> nanofiller may interferes with hydrogen bonding between adjacent urethane polymer chains minimizing sliding between one chains to another as illustrated in Fig.3, since additional peak appears at 1049 cm<sup>-1</sup> due to Si-o-Si and disappearance of N-H at 3336.96 cm<sup>-1</sup>. The results of this study are in agreement with the finding of Qasim et al<sup>(25)</sup>, who found that the mean of flexural strength of VLCR was improved when carbon nanotubes were added.

For impact strength, specimens without notches were selected rather than notched one, since it was time consuming, difficult, impractical and weakened the sample. In addition to that, the brittleness of the material makes notch preparation difficult to be made<sup>(30,31)</sup>. The addition of 2% nanosilica to VLCR significantly decrease the impact strength. Possibly because the (UDMA) stiffness attained by nanofiller addition unfortunately results in more stresses inside the resin matrix especially adjacent to the sharp and irregular borders of SiO<sub>2</sub> nanofiller which might enhance fracture by sudden impact<sup>(32)</sup>, also crack propagation might be enhanced by weak bonding between polymer matrix and nanofiller treated with epoxy coupling agent. In fact, the brittle nature of the material and aggregation of SiO<sub>2</sub> nanofiller inside some pores or voids, which might resulted from polymerization or evaporation of the solvent, could facilitate the formation of micro-cracks within the urethane matrix had weakened the impact strength as revealed by( SEM). In practice, a good balance between impact and flexural strength is desirable because rigid nanocomposite with improved impact strength is needed. Although such a balance is important but positive correlation between such parameter is not always possible in practice. Sometimes one enhanced and the other is deteriorated<sup>(33)</sup>. The finding of this study disagrees with that of Qasim et al<sup>(25)</sup>, who found a significant increase in impact of VLCR when carbon nanotubes were added. This could be due to different material, nanofiller and measuring device.

The surface hardness of the experimental samples were slightly more significant than control groups. This is possibly because of intrinsic

features of SiO<sub>2</sub> nanofiller such as surface hardness beside that, modulus of elasticity and polymerization shrinkage were improved when nano particles were added<sup>(34)</sup>. Moreover, such improvement might be due to fair distribution of SiO<sub>2</sub> nanofiller in polymer matrix which limit deformation under load. Also a strong ionic inter atomic bonding gained by hard SiO<sub>2</sub> nanofiller incorporated into the matrix may enhance some needed properties such as strength and hardness<sup>(35)</sup>.

The non-significant increase in surface roughness of the experimental specimens may be due to slight accumulation of not symmetrical sharp nano particles on sample surface. In addition to that surface roughness was measured by Profilometer which is capable to detect roughness within small micron, thus SiO<sub>2</sub> filler within nano scales might have little effect on this property.

One of the significant properties of acrylic polymer is absorption of water. The polar characteristics of acrylic resin molecules may encourage water sorption slowly through period of time. High amount of water sorption leads to softening of polymer as a result of plasticizing effect that deteriorate the polymer strength<sup>(36)</sup>. In the present study, SiO<sub>2</sub> nanofiller addition significantly decreases the water sorption of the experimental group. The reason could be due to micro voids or pores that encourage water to pass through and out of the polymer resin. SiO<sub>2</sub> nanofiller added might decrease the total volume of water uptakes by acrylic resin because they are insoluble in water<sup>(37)</sup>. The solubility depend on the amount of soluble ingredients from acrylic resin. The initiators, plasticizer, free monomer and some pigments represents the soluble ingredients in acrylic resin<sup>(38)</sup>. The results of the current study shows a non-significant decrease in the values of water solubility of the experimental groups. This is possibly because the addition of insoluble SiO<sub>2</sub> nanofiller to the polymer reduces the total amount of polymer solubility. According to the author knowledge there are no previous studies regarding water sorption and solubility of VLCR reinforced with nanofiller to compare with this study.

This in vitro study showed that VLCR reinforced with 2% SiO<sub>2</sub> nanofiller significantly improve the transverse strength, surface hardness and water sorption. However, this addition lowered the impact strength significantly, suggests further laboratory studies with other nanofiller or modification in mixing technique to achieve higher improvement and better balance between flexural and impact strength.

## CONCLUSION

Within the limitation of this study the following conclusions were drawn:

1. The transverse strength and surface hardness were increased, while the impact strength was decreased when 2% SiO<sub>2</sub> nanofiller was added to VLCR.
2. The addition of 2% SiO<sub>2</sub> nanofiller to VLCR led to a reduction in water sorption and solubility, while non-significant increment in surface roughness was noticed.

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