

# Comparison of Various Properties of a Silorane Based Dental Composite with Two Methacrylate Based Dental Composites

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**Abstract**— The synthesis of a new monomer system named silorane (obtained from the reaction of oxirane and siloxane molecules) gave way to the production of a novel low shrinkage silorane based composite. **Objectives:** The purpose of this study was to compare some physical properties of a silorane based composite (Filtek Silorane, 3MESPE) to those of two well-known low shrinkage methacrylate-based composites (Filtek Supreme XT, 3MESPE; Esthet X HD, Dentsply). The study also includes the effect of external media (food simulating solutions; distilled water, artificial saliva, 25% ethanol, coconut oil and Coke) on the three composites. **Methods:** Cured samples underwent water absorption (with immersion in food simulating solutions) and desorption. The data were analysed; the water absorption and desorption profiles were mapped, the diffusion coefficients and solubility of each sample were calculated. Curing efficiency was measured on the top and bottom surfaces of cured composite samples using Fourier Transform Infrared (FTIR) spectroscopy. Finally, the temperature profile during polymerisation of each composite sample was mapped, allowing the calculation of the sample's maximum exotherm. **Results:** From the results obtained from these experiments, overall, the properties of Filtek Silorane are comparable with those of Filtek Supreme XT and Esthet X HD.

**Index Terms**— absorption, composite resins, immersion, silorane resins

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## I. INTRODUCTION

TO date, modern dental composites exhibit very good physical resistance and aesthetics. The development of the different resins lead to remarkable improvements in terms of physical strength, wear resistance, and stability in the oral environment in general [1]. However, these composites set by a free radical polymerisation reaction involving the carbon double bonds in the dimethacrylate monomers. This contributes to the main shortcomings of dental composites, i.e. marginal leakage due to polymerisation shrinkage [2].

Considerable efforts have been invested to minimize shrinkage stress from polymerisation of dental composite materials. The main approaches adopted so far have been to change the monomer structure or chemistry, respective to change in the filler amount, shape or surface treatment. Methods to modify the monomer matrix have been developed, starting with typical dimethacrylate monomers being modified, for example with urethane dimethacrylates and diluents (e.g. TEGDMA: triethyleneglycol dimethacrylate) [3]-[5]. Other approaches include the development of liquid crystalline monomers or ring-opening systems, to develop non or minimally shrinking dental composites which contain spiroorthocarbonates as additives to dimethacrylate or epoxy-based resins [1], [6]-[9].

Some modern developments in dental composite research have focused on the use of ring-opening systems like oxirane-based resins cured under visible light conditions [2], [10]. Oxirane resins have shown many desirable properties such as improved depth of cure, lower polymerisation shrinkage, higher strength, as well as equivalent hardness and acceptable glass transition temperature when compared with conventional methacrylate based dental composites [10]. However, the in vivo cytotoxicity and mutagenicity of oxirane resins are found to be comparable to those of methacrylate based dental composites [11]. As a result, research continued in the direction of ring-opening monomers, with efforts and focus on reducing the effects of cytotoxicity and mutagenicity.

Recently, Weinmann et al. [2] described the synthesis of a new monomer system for composite resins, named silorane, obtained from the reaction of oxirane and siloxane molecules. The novel resin claimed to have combined the two key advantages of the individual components; low polymerisation shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane [2]. Further, Schweickl et al. [12] showed that the mutagenic potential of various siloranes tested in diverse test systems was much lower than those of related oxiranes [12]. Palin et al. [13] found that the silorane dental composite exhibited significantly lower water sorption, solubility, and associated diffusion coefficient than two established methacrylate based dental composites tested, thus concluded that the decreased water sorption, solubility, and associated diffusion coefficient characteristics of silorane resin may potentially improve the hydrolytic stability of resin-based dental composite restorations [13]. Subsequently, Eick et al. [14] found that silorane resins were stable in simulated biological fluids using aqueous solutions containing either epoxide hydrolase, porcine liver esterase, or dilute hydrochloric acid. These reported advantages enhance the potential of silorane monomers being used successfully as dental composite materials [14].

The purpose of this study was to compare some of the physical properties of a silorane based dental composite to those of two well-known low shrinkage methacrylate based dental composites, and to look at the effect of external media (food simulating solutions; distilled water, artificial saliva, 25% ethanol, coconut oil and Coke) on the three dental composites.

## II. MATERIALS AND METHOD

### A. Materials

Three dental composites that are currently marketed in the United Kingdom as low shrinkage dental composites and five immersion solutions have been studied.

Dental composites (tabulated in Table 1): Filtek Silorane (3MESPE), Filtek Supreme XT (3MESPE) and Esthet X HD (Dentsply).

Immersion solution: Distilled water, artificial saliva (A.S Saliva Orthana, Kastrup, Denmark), 25% ethanol (Poole, England), coconut oil (New Jersey, USA) and Coke (Uxbridge, England).

### B. Methods

1) *Sample preparation:* Disc shaped samples, measuring 6mm in diameter and 2mm thickness were made using pre-prepared silicone moulds. The silicone mould was placed on top of a glass slab covered with an acetate sheet and the cavities were then filled with the appropriate dental composite. The top surface was covered with another acetate

sheet followed by another glass slide. The samples were then cured with a halogen visible light curing unit for 40 seconds, using the standard dental composite cure following the manufacturers' recommendations. The samples were inspected for flaws. Any samples with visible voids or flaws were discarded.

2) *Absorption and desorption studies:* Five samples of each of the three dental composites underwent a period of preconditioning; they were placed in a drying oven at  $37 \pm 1^\circ\text{C}$  for at least 1 day. Then each sample was weighed to an accuracy of 0.0001g, using an analytical microbalance and placed in individual screw top, glass storage jars containing 100ml of each appropriate immersion solution and stored in an oven at  $37 \pm 1^\circ\text{C}$ . At noted intervals each sample was taken out of the glass storage jar, blotted on filter paper to remove excess water, weighed and returned to the bottle. On day one, weight measurements were taken at 5, 10, 20, 40, 60 minutes, and then every hour for the next 6 hours, with subsequent measurements taken once to twice a day thereafter. The water uptake was recorded until there was no further change in weight i.e. equilibrium had been reached (constant weight or within  $\pm 0.0002\text{g}$ ).

Once the samples had equilibrated, the samples were desorbed in a drying oven kept at  $37 \pm 1^\circ\text{C}$ . Again, as for the absorption studies, the samples were weighed at noted intervals, until equilibrium was reached (weight remained constant or within  $\pm 0.0002\text{g}$ ).

3) *Calculating diffusion coefficient:* The diffusion coefficients,  $D$ , for absorption and desorption were calculated for each sample using (1):

$$D = s^2 \pi (4L^2) / 16M \infty 2 \quad (1)$$

Where  $4L$  is the thickness of the sample (in meter).

4) *Calculating solubility:* The percentage weight change (solubility) of each sample was calculated using (2):

$$\% \text{ Solubility} = [(W_o - W_d) / W_o] \times 100 \quad (2)$$

Where  $W_o$  is the original weight of the sample prior to placement in any solvent, and  $W_d$  is the final dehydration weight of the sample.

5) *Measuring degree of conversion:* 10 samples were prepared for each type of dental composite. These were left for at least 10 minutes (to ensure polymerisation was near complete). The bottom and the top surfaces of the sample were noted. Each surface in turn was pressed against the analysing crystal with a torque wrench device. The spectra were acquired, employing a micro-MIR (multiple internal reflection) cell attached to a Fourier-Transform Infrared (FTIR) spectrometer with the following conditions; scan range 2000 – 600  $\text{cm}^{-1}$ , resolution 4  $\text{cm}^{-1}$ ,  $45^\circ$  paraedge KRS-5 minicrystal of 7 internal reflections, 10 scans per sample, at ambient temperature.

Uncured composite resins ( $n=10$ ) were also placed in the

spectrometer and scanned using the same scanning conditions.

For the methacrylate based dental composites (Filtek Supreme XT and Esthet X HD), measurements of the peak heights were undertaken using the stretch vibrations of the methacrylate group, aliphatic C=C bonds at 1635 cm<sup>-1</sup>. For the silorane based dental composite (Filtek Silorane), measurements of the peak heights were undertaken using the stretch vibrations of the epoxy rings, C-O-C at 884 cm<sup>-1</sup>. The stretch vibrations of the aromatic C=C group at 1608 cm<sup>-1</sup>, were used as a reference frequency for both methacrylate based and silorane based dental composites. Ratios of cured to uncured samples were calculated, giving an indication of the degree of conversion (i.e. 100% conversion would give a result of infinity, ∞; Equation 3 and 4). This protocol was adapted from Palin et al., 2005 and Papadogiannis et al., 2009 [15, 16].

Methacrylate based dental composite degree of conversion (%C=C)

$$= \frac{\text{Aliphatic (C=C) / aromatic (C=C) cured}}{\text{Aliphatic (C=C) / aromatic (C=C) uncured}} \times 100 \quad (3)$$

Silorane based dental composite degree of conversion (%C-O-C)

$$= \frac{\text{Epoxy ring (C-O-C) / aromatic (C=C) cured}}{\text{Epoxy ring (C-O-C) / aromatic (C=C) uncured}} \times 100 \quad (4)$$

6) *Measuring polymerisation exotherm:* The thermal emission during the dental composites' polymerising was measured by a K-type thermocouple and a digital thermometer. The thermocouple was secured in a groove on an acrylic based plate, so that the surface of the thermocouple was flush with the top of the base plate. A silicone mould was placed over the thermocouple, measuring 6mm diameter and 2mm thickness. The experimental setup allowed the thermocouple to be placed at the centre of the 6mm diameter hole. The dental composite material was placed in the mould, and a 1mm glass plate was placed over the sample. The sample was then cured for 40 seconds using the light curing unit, holding the light gun directly on the glass plate. The temperature was noted before curing commenced (at 0 second), at the maximum exotherm during light curing (within the 40 seconds cure), and 20 seconds after light curing ended (at 60 seconds). 10 samples for each composite type were tested.

7) *Statistical methodology:* SPSS version 12 was used to analyse the raw data, calculating means, standard deviations, and confidence intervals at the 95% level. Statistical significant testing was undertaken using a one-way ANOVA with post hoc tests (p value at 0.05). Microsoft Excel 2007 was used to plot the graphs. The data was analysed statistically, graphically and descriptively.

### III. RESULTS

Table I: Dental composites used in this study.

TABLE I  
MANUFACTURERS DETAIL OF DENTAL COMPOSITE USED IN THIS STUDY

Dental composite / Manufacturer	Lot Number / Expiry date	Type	Filler	Resin Matrix
Filtek Supreme XT 3M ESPE	N110380 08/2012	Nano hybrid methacrylate based	Silica nanofiller, zirconia/silica nanocluster, 78.5wt%	Bis-GMA, Bis-EMA, UDMA, TEGDMA
Esthet X HD Dentsply	090430 04/2012	Nano hybrid methacrylate based	Ba-F-Al-B-Si-glass (particle size <1µm), silica nanofiller (0.04µm), 77wt%	Bis-GMA adduct, Bis-EMA adduct, TEGDMA
Filtek Silorane 3M ESPE	N125012 07/2011	Micro hybrid silorane based	Silanized quartz, ytterbium trifluoride, (inorganic fillers 0.1 - 2µm), 76 wt%	Epoxy cyclohexylethyl cyclopolymethylsiloxane, Bis-3,4-epoxycyclohexylethylphenylmethylsilane

Table II: Maximum water absorption data for Filtek Supreme XT, Esthet X HD and Filtek Silorane immersed in distilled water, artificial saliva, 25% ethanol, coconut oil and Coke.

TABLE II  
EQUILIBRIUM WATER ABSORPTION DATA ON THE THREE DENTAL COMPOSITES

Dental Composite	Solvent	Water Absorption									
		Distilled water		Artificial saliva		25% ethanol		Coconut oil		Coke	
		Max %water uptake	*Time (days)	Max %water uptake	*Time (days)	Max %water uptake	*Time (days)	Max %water uptake	*Time (days)	Max %water uptake	*Time (days)
Filtek Supreme XT		≤1.5	26	≤1.5	26	≤1.5	26	-	-	≤1.5	26
Esthet X HD		<1.0	33	<1.0	33	<1.0	33	-	-	≤0.5	11
Filtek Silorane		<1.0	72	<1.0	72	<1.0	72	-	-	<1.0	72

\*Time to equilibrate.

Table III: Equilibrium water desorption data of Filtek Supreme XT, Esthet X HD and Filtek Silorane after immersion in distilled distilled water, artificial saliva, 25% ethanol, coconut oil and Coke.

TABLE III  
EQUILIBRIUM WATER DESORPTION DATA ON THE THREE COMPOSITES

Dental Composite	Solvent	Water Desorption									
		Distilled water		Artificial saliva		25% ethanol		Coconut oil		Coke	
		Max %water loss	*Time (days)	Max %water loss	*Time (days)	Max %water loss	*Time (days)	Max %water loss	*Time (days)	Max %water loss	*Time (days)
Filtek Supreme XT		≤1.5	7	≤1.5	7	≤2.0	7	-	-	≤1.5	7
Esthet X HD		<1.0	14	<1.0	14	<1.0	14	<0.6	4	<0.6	4
Filtek Silorane		≤0.6	8	≤0.6	8	≤0.6	8	≤0.5	8	≤0.6	8

\* Time to equilibrate.

Table IV: Diffusion coefficient values obtained for absorption and desorption for the three dental composite materials in distilled water, artificial saliva and Coke.

TABLE IV

SUMMARY OF DIFFUSION COEFFICIENTS ( $10^{-12}m^2sec^{-1}$ ) FOR ABSORPTION AND DESORPTION (IN PARENTHESES), FOR THE THREE DENTAL COMPOSITES

Medium	Filtrek Supreme XT	Filtek Silorane	Esthet X HD
Distilled Water	1.75 (3.68)	0.42 (2.54)	2.24 (4.66)
Artificial Saliva	1.11 (1.62)	0.56 (9.94)	0.92 (2.85)
25% Ethanol	1.36 (3.59)	0.23 (2.55)	3.25 (3.14)
Coke	1.57 (9.37)	0.82 (2.41)	1.77 (8.00)
Coconut Oil	0 (52.90)	0 (26.40)	0 (45.73)

Fig. 1: The degree of conversion at the top surface and bottom surface of cured Filtek Supreme XT, Esthet X HD and Filtek Supreme XT (95% confidence interval marked on each composite group).

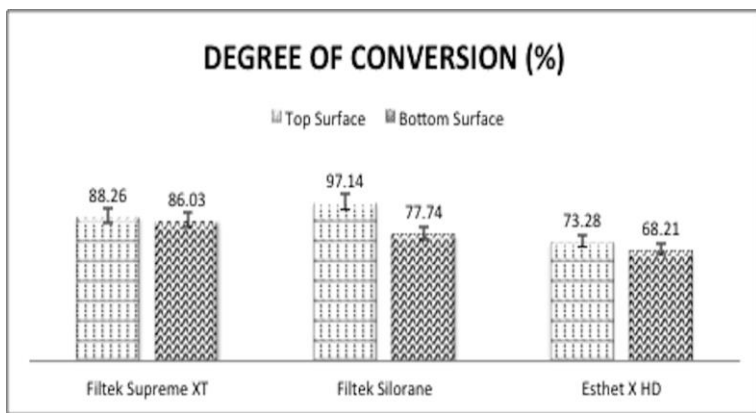
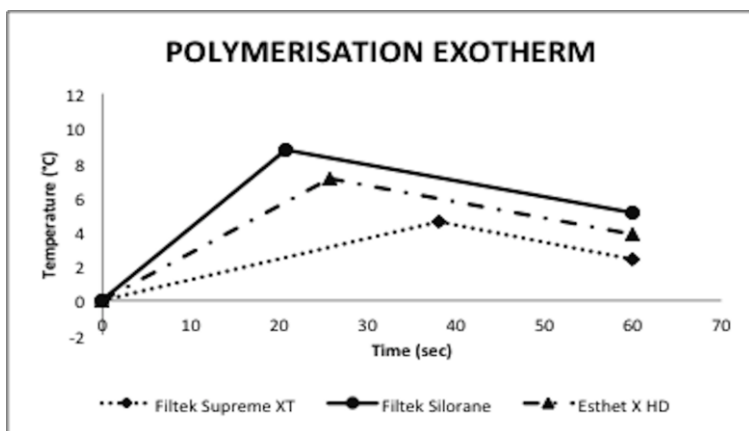


Fig. 2: Polymerisation exotherm of Filtek Supreme XT, Esthet X HD and Filtek Silorane.



IV. DISCUSSION

From Table 2 it can be concluded that the water absorption profiles were similar for Filtek Supreme XT and Esthet X HD immersed in distilled water, artificial saliva, 25% ethanol and Coke. Variations were only seen in the maximum water uptake and the time taken to reach equilibrium; Filtek Supreme XT,  $\leq 1.5\%$  in the first 26 days, Esthet X HD,  $< 1.0\%$  in the first 33 days. After this, the water uptake slowed down, indicating that the samples were reaching equilibrium. However, Filtek Supreme XT in distilled water and Esthet X HD in Coke began to lose weight after reaching their maximum water uptake ( $\leq 1.5\%$  in the first 26 days, and  $\leq 0.5\%$  in the first 11 days) indicating that as well as absorbing water, the dental composites were leaching constituents. Filtek Silorane in distilled water, artificial saliva, 25% ethanol and Coke, absorbed water linearly and very slowly, throughout the entire duration of this experiment (~ 72 days). The average maximum water uptake in these solvents was  $\leq 1.0\%$ . There was no weight change (water uptake) seen in all the dental composites immersed in coconut oil. This would be expected since the latter is a very hydrophobic material, essentially a paraffin, that is completely immiscible in water. To summarise, the water uptakes of the three dental composites in the five media is classed as low.

From Table 3 it is clear that Filtek Supreme XT and Esthet X HD immersed in distilled water, artificial saliva and Coke had low weight losses, with differences only seen in the maximum weight loss and the time taken to reach equilibrium; Filtek Supreme XT,  $\leq 1.5\%$  in the first 7 days; Esthet X HD,  $< 1.0\%$  in the first 14 days. These results coincide with the water uptake results above. A different result was seen with the percentage loss from Filtek Supreme XT in 25% ethanol; the maximum loss was  $\leq 2.0\%$ , which was more than the percentage water uptake. Esthet X HD in coconut oil appeared to lose weight even though no water uptake was seen before. Filtek Silorane immersed in distilled water, artificial saliva, 25% ethanol and Coke, had a maximum loss of  $\leq 0.6\%$ , which is lesser than the water uptake. Also, equilibrium was reached in the first 8 days. Filtek Silorane in coconut oil, also appeared to have a small weight loss ( $< 0.5\%$ ) during desorption, even though there was no uptake observed, presumably the materials extracted were organic and at least partially soluble in coconut oil. To summarize, there was very little difference in weight change of the Filtek Silorane compared to Filtek Supreme XT and Esthet X HD during desorption.

The overall diffusion coefficients obtained for both the absorption and desorption processes (Table 4) are in the general range for dental composites [17]. It is clear that the values for desorption are generally much higher than those in absorption. This is symptomatic of the water clustering around hydrophilic sites in the dental composite [18]. The desorption diffusion coefficients for the three dental

composites in coconut oil are much larger than those obtained in other aqueous media, where water is the desorbing species ( $p < 0.001$ ). Hence, it can be assumed that molecules other than water are being desorbed in coconut oil. It is not clear at this stage what these are.

This study found that there was no significant difference in solubility between the dental composites immersed in all the solvents used in this study, indicating that the amount of leachant from all the dental composites was the same. For each sample, small amounts of material being absorbed/released by the dental composites, could not be detected by gravimetric weight measurements, using a four figure balance. After averaging the gains/losses of the five samples, these cumulative results could occur. Thus, the differences in solubility between each sample were so small that they were insignificant.

To conclude, there was very little difference in weight change of the Filtek Silorane compared to Filtek Supreme XT and Esthet X HD observed in the absorption and desorption studies. The overall diffusion coefficients obtained for these three dental composites are in the general range for dental composites [17] and there was no significant difference in solubility of these three dental composites that can be observed in this study. Palin et al. [13] found that the silorane based dental composite at 26 days of immersion in distilled water, exhibited significantly lower water sorption solubility and associated diffusion coefficient, than the established methacrylate based dental composites tested. Their samples were disc shaped, but larger in diameter (12mm x 2mm) compared to those used in this study (6mm x 2mm), which could have affected the inconsistencies in water sorption, solubility and diffusion coefficient values.

Figure 1 shows that there were no significant differences on the degree of conversion at the top and bottom surfaces of cured Filtek Supreme XT and Esthet X HD. However, the degree of conversion of Filtek Silorane at the top surface was significantly higher than the bottom surface ( $p = 0.001$ ). This indicated that more monomer was converted to polymer at the top surface of Filtek Silorane than the bottom surface. Clinically, this result implicates that more free monomer will be present at the bottom surface of a cavity restored with Filtek Silorane. This can result to potential cytotoxicity effect to the pulp and the potential loss of structural bonding of the tooth structure to the dental composite material. Comparisons between the three dental composites show that the degree of conversion at the top surface of Filtek Silorane is significantly higher than Filtek Supreme XT ( $p = 0.041$ ) and Esthet X HD ( $p = 0.003$ ). However, the degree of conversion at the bottom surface shows that the Filtek Supreme XT has the highest percentage conversion, followed by Filtek Silorane ( $p = 0.046$ ) and Esthet X HD ( $p = 0.012$ ). Taking all this into consideration, it can be assumed that the overall degree of conversion of Filtek Silorane was higher than the Filtek Supreme XT and Esthet X HD. Papadogiannis et al. [16] tested the curing efficiency of low shrinking

methacrylate based dental composites (Ceram X Mono, Premise, Clearfil Majesty) and the silorane based dental composite (Filtek Silorane), using an ATR-FTIR spectroscopy. They found that Filtek Silorane exhibited the highest degree of conversion at the top surface. Also, all the tested dental composites exhibited a lower degree of conversion at the bottom surface in general. This finding is in line to the finding of this current study.

Figure 2 shows that Filtek Silorane reached maximum exotherm faster (~20 seconds), followed by Esthet X HD (~30 seconds;  $p < 0.001$ ) and Filtek Supreme XT (~40 seconds;  $p < 0.001$ ). However, the maximum exotherm of Filtek Silorane was the highest ( $\leq 10^\circ\text{C}$ ), followed by Esthet X HD ( $\leq 8^\circ\text{C}$ ;  $p < 0.001$ ) and Filtek Supreme XT ( $\leq 5^\circ\text{C}$ ;  $p < 0.001$ ). The maximum exotherm of Filtek Silorane observed in this study was definitely higher than the proposed tolerance threshold for tooth pulp ( $7-8^\circ\text{C}$ ) [19]. However, the test to observe the relation of this polymerisation exotherm to the temperature increase in tooth pulp chamber was not carried out in this study. It would seem that further investigations, both on the physical properties of the new Filtek Silorane dental composite and on the in vivo temperature increase during polymerisation are needed. The effect of heat abstraction through the layers of tooth connective tissue should also be considered. Dabrowski et al. [20] found that Filtek Silorane showed a significant temperature increase during polymerisation compared to two other methacrylate based dental composites. They found that Filtek Silorane maximum exotherm was  $24^\circ\text{C}$  (when cured using a halogen lamp) and  $32^\circ\text{C}$  (when cured using a diode lamp), compared to Filtek P60 (halogen lamp:  $9^\circ\text{C}$ ; diode lamp:  $10^\circ\text{C}$ ) and Valux Plus (halogen lamp:  $9^\circ\text{C}$ ; diode lamp:  $12^\circ\text{C}$ ). They concluded that the different course of polymerisation reaction of Filtek Silorane (the cationic ring opening polymerisation) was the cause of the increased temperature. They also found in their clinical observation, that the high temperature produced by Filtek Silorane during polymerisation did not show any negative reaction to the tooth pulp, hypersensitivity after treatment, or negative patient experience after treatment. Miletic et al. [21] investigated the temperature changes during curing of Filtek Silorane (silorane based), Admira (ormocer based) and Herculite XRV (methacrylate based) dental composites, at the bottom surface of the dental composites and in the teeth pulp chamber roof dentin. They observed that there was a substantially higher temperature rise in Filtek Silorane ( $45^\circ\text{C}$ ), compared to the other two materials (Admira:  $27^\circ\text{C}$ ; Herculite XRV:  $29^\circ\text{C}$ ). However, it was also observed that there was no difference in the temperature rise inside the pulp chamber. They suggested that this was probably due to the insulating effect of the remaining dentine of the cavity.

## V. CONCLUSIONS

This study concludes that the properties of Filtek Silorane are comparable with those of Filtek Supreme XT and Esthet X HD. However, we strongly believe that the new silorane based dental composite could potentially replace the methacrylate based dental composites used in dentistry in the near future due to its low polymerisation shrinkage property and its independence from Bisphenol A (a carcinogenic material which are widely used in methacrylate based dental composites [9]). It would seem that further investigations, both on the physical properties of the silorane based dental composite and on the in vivo effect of these properties are needed.

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