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Evaluation of Physical Properties of Silica filled Resin Based Dental Composites

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Abstract*: In this study, resin based dental composites were developed using monomers BisGMA and TEGDMA, CQ and DMAEA. Silica were filled as filler material. After curing, the samples were tested for void content and water sorption and solubility. The results showed that as the void content increases, water sorption also increases in resin based dental composites. In brief, it can be stated through this experimental study that with the increase in silica filler upto 9 %wt, void content and water sorption both increases.

***Keywords* : Dental Composites, Resin etc.**

I. INTRODUCTION

Resin-based dental composites are the most commonly used restoration substance for restoration of teeth because of fine esthetics and their compatibility with tooth [1, 2]. The physical, mechanical, optical and tribological properties of resin-based composites are also very impressive and suitable to produce a durable product that can sustain itself in human oral environment. Resin based dental composites generally comprise of monomers [3]. Bisphenol A glycidyl methacrylate (Bis-GMA) is one of the most commonly used resin [4]. The superior aesthetic quality of Bis-GMA and its suitable mechanical properties make them an ideal option to use as a resin base. However, its high viscosity makes it difficult for introducing and mixing filler particles into it. To counter this, Tri-ethylene-glycol-dimethacrylate (TEGDMA) was used along with Bis-GMA to lower the overall viscosity of the resin base [5]. This ensures the smooth introduction and mixing of the filler particles and other reactive components resulting in uniform and consistent distribution of mixed components. The resin matrix needs UV light for its polymerization. Ultra Violet rays are introduced to initiate the polymerization of the mixture. Photo initiators are used to trigger the photo polymerization process such as Camphorquinone (CQ). It is the most commonly used photo initiator due to its good clinical acceptance [6].

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It adds a slight yellow tint to the uncured composite mixture, although this colour bleaches during curing.

The photo initiators produce free radicals when exposed to radiation. CQ absorbs radiation due to $\pi \rightarrow \pi^*$ transition of the α -dicarbonyl chromophore and produces an excited singlet state which further goes to an excited triplet state and reacts with oxidizable species and decomposing into colourless product in the process [7]. Due to light absorbing photo initiator being used, there is an inevitable decrease in absorbing property of the initiator with increase in illumination time. This process is called photobleaching and this often hinders the curing of thick samples. Ceramic fillers are generally used to improve mechanical properties of dental composites [8]. Silane treatment of filler particles is done to enhance the bonding between filler and the resin matrix [9].

Voids are considered as important characteristic of resin based dental composites [10]. In composites, high viscosity of resin is one of the major reason of void content. Resin matrix does not penetrate completely in the void spaces between the filler particles and hence causes voids. When filler particles are tightly packed, void content generally increases.

Preventing these voids becomes a more daunting task when the fillers/fibers are packed tightly together in a composite. Water sorption and solubility is also an important property of dental composite which affects its performance [11]. The objective of this research is to study the effect of silica filler particles on void content along with water sorption and solubility of resin based dental composite.

II. MATERIALS AND METHODS

In this study, Bis-GMA (Sigma Aldrich) and TEGDMA (Tokyo Chemical Industry, Japan) were used as resin monomers for matrix. Camphorquinone (Spectrochem Pvt. Ltd., Mumbai) was used as a photo initiator. Dihydroxy ethyl-para-toluidine (DMAEA) (Tokyo Chemical Industry, Japan) was used as an accelerator in the process which is an unsaturated carboxylic acid ester containing a tertiary amino group. It is water miscible liquid with pungent amine like odour and has colourless to yellowish colour. It is colourless to yellowish, water miscible liquid with a pungent amine-like odour. Micro Silica powder was used as filler for the composite. It is an inorganic compound. SiO2 is a non-toxic white powder that is insoluble in water. Silane treatment of silica particles was carried out in order to enhance the bonding between resin and filler particles.

3-(Trimethoxysilyl)propyl Methacrylate (TCI Chemicals (India) Pvt. Ltd.) was used as a silane coupling agent. For silane treatment, 95% ethanol/5% water solution was mixed with acetic acid to make the pH 4.5–5.5 solution. Then silane was added with stirring to yield a 2% final concentration. Solution was left for five minutes for hydrolysis and silanol formation. The filler particles were submerged in the solution and gently agitated and removed after 2-3 minutes and then decanting the solution. The filler particles were briefly rinsed twice with ethanol and dried for 5–10 minutes at 110°C. LED Light Curing Unit (1200 mW/cm2, 450 to 490 nm, Dentmark, Mumbai, India) was used to carry out the curing process. Curing was done on the samples for 20 seconds.
The samples were prepared as per the composition given in Table 1.

Table 1. Composition of Dental Composites

S. No.	Sample Designation	Composition
1	RBDC-0	Resin + Filler (0% Silica)
2	RBDC-3	Resin + Filler (3% Silica)
3	RBDC-6	Resin + Filler (6% Silica)
4	RBDC-9	Resin + Filler (9% Silica)

III. METHODOLOGY

Void Content Test

Void content is the measurement of void spaces in composites which reduces the strength of composite significantly. For the measurement of void content, theoretical densities of both the resin and the reinforcing material is required to determine the theoretical density of composite [12]. The individual densities are normally obtained from the supplier of the resin and reinforcing material. The measured theoretical density of the material is calculated using equation 1.

$$\rho_{th} = \frac{W_1 + W_2 + W_3 + W_4 + W_5}{\frac{W_1}{\rho_1} + \frac{W_2}{\rho_2} + \frac{W_3}{\rho_3} + \frac{W_4}{\rho_4} + \frac{W_5}{\rho_5}} \quad (1)$$

Where; ρ1, ρ2, ρ3, ρ4 and ρ5 are densities for different-different materials.
Apparent density of composite can be calculated using Archimedes principle of water immersion test. After calculating theoretical and apparent density, void content of composite can be calculated using equation 2.

$$\text{Void Content} = \frac{\text{Theoretical density} - \text{Apparent density}}{\text{Theoretical density}} X\ 100 \quad (2)$$

Water Sorption and Solubility

Water sorption and solubility tests were performed as per ISO 4049 standards [13]. After curing process, the thickness of cured sample was measured at 5 different sections using digital calipers. These readings were averaged to find the mean diameter of the specimen usinto further calculate its volume.

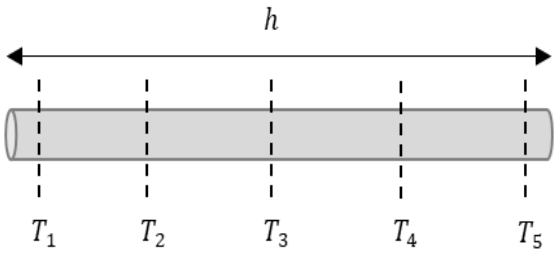

Figure 1. Dental Composite Specimen

Specimens are stored in a desiccator at 37±1℃ with silica gel for 22 hours and then at 23±1° for 2 hours. The specimen is weighed once the mass has stabilized (dry mass M1). Specimen were stored in distilled water for 30 days. After 30 days, specimens were taken out, wiped gently and weighed (wet mass M2). After this, the specimen was stored in desiccator at 37℃ and weighed till constant mass has been achieved (dry mass M3). All weight measurements were done using an analytical balance of 0.1 mg accuracy (AUW220D; Shimadzu Corp., Kyoto, Japan)
Water sorption and Solubility were calculated using equations 3 and equation 4, respectively.

$$Water\ Sorption\ (WS) = \frac{M_2 - M_3}{V} \quad (3)$$

$$Solubility\ (SL) = \frac{M_1 - M_3}{V} \quad (4)$$

Where, M1 is the initial dry mass, M2 is the final wet mass and M3 is the final dry mass.

IV. RESULTS, DISCUSSION AND CONCLUSION

Void content of silica filled resin based dental composite were calculated as per ISO 4049 standard discussed in methodology section. The experimental results of void content tests are tabulated in Table 2.

Table 2. Void Content Volume of Resin Based Dental Composite

Sr. No.	Sample Designation	Void Content (%)
	RBDC-0	0.56
1	RBDC-3	0.84
2	RBDC-6	1.25
3	RBDC-9	1.58

Void content results shows that with the increase in silica filler %wt, void content increases.
Water sorption and solubility of silica filled resin based dental composites were evaluated as per the methodology discussed in Methodology section.
The experimental results of water sorption and solubility are tabulated in Table 3.

Table 3. Experimental Results for Water Sorption and Solubility

Sample Designation	Water Sorption (µg/mm^3)	Solubility (µg/mm^3)
RBDC-0	1.86	0.091
RBDC-3	2.05	0.14
RBDC-6	2.34	0.18
RBDC-9	2.57	0.98

From the above table it can been concluded that water sorption increases with increase in filler loading. These results reflects the consequence of trends of void content. Void in composite may provide space for water sorption. So, as the void content increases, water sorption also increases in resin based dental composites. In brief, it can be stated through this experimental study that with the increase in silica filler upto 9 %wt, void content and water sorption both increases. As per ISO 4049, the values of water sorption and solubility should be le less than 40 µg/mm3 and 7.5 µg/mm3, respectively. The results of this study are in fine agreement of ISO 4049 standards.

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