

Long-term solubility and sorption characteristics of novel dental restoratives

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Abstract

Sorption and solubility are the unfavorable conditions for the dental restorative composites (DRCs). It can be precursor of various physical and chemical phenomenon that may lead to structural deterioration and minimizes the endurance of restorations. This study sought to evaluate the sorption and solubility features of MPTS (M)/APTES(A) treated n-HAPs filled dental composite in distilled water and artificial saliva medium. In this experiment, 7 different compositions of disc-shaped specimens of $\Phi 15\text{mm} \times 1\text{mm}$ ($n=3$) of dental composites were prepared and tested under distilled water and artificial saliva medium for 35 days according to ISO 4049 method. The dental material shows relatively higher sorption and solubility in the artificial saliva medium as compared to distilled water. However, at higher wt.% (DRCs-12M & DRCs-12A), it shows minimum solubility (i.e.) and sorption characteristics. The results indicate that there was significant variation regarding storage medium and time to saturation but still these values are within the limit of the ISO 4049 standards, which is $40 \mu\text{g}/\text{mm}^3$ for sorption and $5 \mu\text{g}/\text{mm}^3$ for Solubility.

Keywords: Restorative materials, APTES/MPTS treatment, n-HAPs fillers, Sorption, Solubility.

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1. Introduction

Dental Restorative Composites (DRCs) are the radically used dental materials and having much clinical significance because of their revolutionary aesthetics and good adhesive bonding to dental structure (Silva, et al., 2017; Mitra, et al., 2003). The clinical progress of these materials greatly depends on distinct factors i.e. physical, chemical, mechanical, properties and its biological effects on dental pulp as well as soft tissues, water sorption and solubility features of restoratives and still shows a challenging field of dental practice (Eisenburger, et al., 2003). The advancements in these properties have led to extensive versatility and application of dental restoratives for both posterior and anterior teeth (Sabatini, 2015). Under oral environment, these dental restoratives are subjected either continuously or intermittently to chemicals found in food, drinks and saliva (Yap, et al., 2001). Literatures have shown that beverages and some dietary foods i.e., generally acidic in nature may cause surface deterioration of dental restoratives (McKenzie, et al., 2003; Hengtrakool, et al., 2011). Even after an efficient polymerization, restorative

composites show instability and may interact with oral fluids by absorbing water/ oral fluids and releases chemical substances in the oral cavity (Leal, et al., 2017; Sideridou, et al., 2003).

The phenomena of sorption and solubility characteristics are negative in nature and can be precursor of various physical and chemical phenomena that may lead to structural deterioration and minimizes the endurance of restorations. Water sorption may cause volumetric expansion of restoratives that may compensate for polymerization shrinkage, physical and chemical changes such as plasticizing, oxidation and hydrolysis (Ferracane, 2006). However, it is related to the inferior mechanical properties negotiating with the ability of adhesion bonding and subsequently leakage of carcinogenic bacteria's and saliva via restoratives -tooth interface. The metabolism activities of microorganisms produce acids i.e., responsible for lowering the pH of mouth which leads to pulp injury, recurrence of dental caries, discoloration and the problem of tooth hyper-sensitivity (Heshmat, et al., 2013; Mese, et al., 2008; Gerdolle, et al., 2008; Zhang & Xu, 2008). On the other hand, Solubility is decomposition or dissolution of restoratives at specific temperature over a period in oral fluids/ saliva that can be quantified as weight loss per unit volume (Powers & R., 2006). In addition, interaction of some organic constituents of restorative composites with body may cause systemic and local allergic reactions (Kanchanasavita, et al., 1997; Cefaly, et al., 2006).

DRCs are usually classified as per the filler type, size and contents such as nanohybrid, micro-hybrid, macrofill and microfill. Most of the dental composites organic phases consist of methacrylate-based monomers, such as UDMA, BisGMA and TEGDMA, dispersed phases, photo-initiator and co-initiator system including coloring pigments and stabilizers (Kalachandra, et al., 1997; Sideridou, et al., 2002). Silorane based dental restoratives (obtained from the siloxane and oxirane molecules) are the alternative to methacrylate-based resin composites characterized by hydrophobicity, low polymerization shrinkage (Arocha, et al., 2013). Literature studies reveals that silorane based dental restoratives shows decreased sorption and solubility along with improved mechanical properties as compared to traditional methacrylate based dental composites (Eick, et al., 2006; Ilie & R., 2009; Weinmann, et al., 2005). Thus, considering the effect of oral environment on dental restoratives, this paper represents the experimental analysis of sorption and solubility characteristics of MPTS/ APTES treated n-HAPs filled DRCs in distilled water and artificial saliva medium.

2. Materials and Methods

2.1 Materials:

The seven different composition of dental restorative composites were prepared for this study as mentioned in Table 1. These experimental DRCs were prepared with organic methacrylate-based monomers consisting Bis-GMA (bisphenol A glycidyl methacrylate); HEMA (2-hydroxyethyl-methacrylate), TEGDMA (Triethylene glycol-dimethacrylate), Camphorquinone (CQ) and DMAEMA (di-methylamino ethyl methacrylate), and inorganic phase of nano-hydroxyapatite (n-HAPs) particles (20-80nm size), Silane additives i.e. MPTS(3-methacryloxypropyl) trimethoxy silane) and APTES (3-aminopropyl) triethoxysilane) were used as coupling agents. All these analytical grade materials were procured from Sigma Aldrich USA (Bis-GMA, CQ and DMAEMA), TCI Japan (TEGDMA, HEMA and silane additives) and n-HAPs was procured from Nano Research Lab Jamshedpur, Jharkhand.

2.2 Procedure of Silane functionalization of nanohydroxyapatite fillers:

The surface functionalization of n-HAPs particles was performed with 5 wt. % of MPTS and APTES silane (relative to n-HAPs particles) and 70:30 acetone-water solution. The pH of the mixture was maintained to 4 with the help of 3 M acetic acid solution and sonicated for 1 for getting homogeneous mixing with hydrolysis and silanol formation. After that n-HAPs fillers were added in the solution and stirred for approximately 2 hours at 40°C (condensation and hydrogen bond formation). The final mixture was filtered and washed with acetone and then dried in vacuum oven at 50°C for 24 hours that leads to the development of strong covalent bonds between an inorganic substrate and silanol.

2.3 Preparation of Experimental Dental Restorative Composites:

The methacrylate-based monomers Bis-GMA, HEMA and TEGDMA were mixed uniformly for 1 hour at 40°C. After that, MPTS and APTES treated n-HAPs were added into the mixture respectively and again the mixture was stirred for 2 hours. To make it photocurable, CQ and DMAEMA were added into the mixture and container was fully covered with aluminum foil to reduce the photo-initiated polymerization. Seven different DRCs were prepared with 0, 4,8, and 12 wt.% as per given compositions in Table 1. The prepared mixture was then poured into the mold and cured with LED light activation curing unit. All fabricated samples were kept in distilled water at 37°C for 24 hours to complete the polymerization process and dried in the vacuum oven to remove the excess water from the samples.

Table 1. Composition for the organic and inorganic phases of Experimental Dental Restorative Composites

Sample Formulations	Organic Phase					Inorganic Phase	
	Bis-GMA (Wt.%)	TEGDMA (wt.%)	HEMA (wt.%)	CQ (wt.%)	DMAEMA (wt.%)	MPTS treated n-HAPs(wt.%)	APTES treated n-HAPs(wt.%)
DRC-0	60	19.5	19.5	0.2	0.8	-	-
DRC-4M	60	19.5	19.5	0.2	0.8	4	-
DRC-8M	60	19.5	19.5	0.2	0.8	8	-
DRC-12M	60	19.5	19.5	0.2	0.8	12	-
DRC-4A	60	19.5	19.5	0.2	0.8	-	4
DRC-8A	60	19.5	19.5	0.2	0.8	-	8
DRC-12A	60	19.5	19.5	0.2	0.8	-	12

2.4 Solvent Uptake Measurement

Water and artificial saliva sorption/solubility were determined in order to evaluate the hydrophobicity of experimental dental restorative composites that indirectly associated to the nature and volume fraction of organic contents (polymer matrix and silanes). In this experiment, 7 different compositions (Table 1) of disc-shaped specimens with $\Phi 15\text{mm} \times 1\text{mm}$ ($n=3$) of dental composites were prepared {earlier explained in section II B} and tested under distilled water and artificial saliva medium for 35 days according to ISO 4049 method. Similar to Randolph et al. (Randolpha, et al., 2016) and Sideridou et al. (Sideridou, et al., 2011), initially both the samples were weighted with weighing balance of precision 0.0001 mg (i.e., m_o) and immediately kept in distilled water and artificial saliva for 7 days at $37 \pm 1^\circ\text{C}$. At the interval of one week all the samples were removed from the storage medium, blotted dry and again weighted (i.e., m_w) and after that again dipped in the solutions. When all the samples get saturated (absorbs no more solvent) then these were kept in vacuum oven to make it completely dry. All the samples were regularly weighted till the measured value stabilized and final weights were measured (i.e., m_d). The volume of sample was termed as V . The solvent sorption and solubility i.e., released matter of DRCs were determined in (%) by using Eqns. (1) & (2) and in $\mu\text{g}/\text{mm}^3$ by using Eqns. (3) & (4).

$$W_{SP}(\%) = \frac{m_w - m_o}{m_o} \quad (1)$$

$$W_{SI}(\%) = \frac{m_d - m_o}{m_o} \quad (2)$$

$$W_{SP} \left(\mu \frac{\text{g}^3}{\text{mm}} \right) = \frac{m_w - m_d}{V} \quad (3)$$

$$W_{SI} \left(\mu \frac{\text{g}^3}{\text{mm}} \right) = \frac{m_o - m_d}{V} \quad (4)$$

Table 2. Chemical Composition of Artificial Saliva

S. No	Chemical Compound	wt.% /l distilled water
1.	Sodium carboxymethyl cellulose (CMC)	1.0000
2.	Potassium chloride (KCl)	0.1200
3.	Sodium chloride	0.0844
4.	Calcium chloride (CaCl ₂)	0.0146
5.	Magnesium chloride (MgCl ₂)	0.0052
6.	Potassium dihydrogen phosphate (KH ₂ PO ₄)	0.0342
7.	Sorbitol 70% solution	3.0000

3. Results and Discussion

Under the oral environment, dental restorative composites are exposed to various physical and chemical agents present in drinks, saliva, food and other oral hygiene routines. These composites should be resistant to different oral environments and show minimum diversity inside the mouth because interaction between these agents may lead to degradation of restorative composites (Akova, et al., 2006). The Artificial saliva used in this experiment was selected depending on its viscosity and pH like natural human saliva that have more influence on the diffusion phenomenon in restorative composites than that of distilled water (Darvell, 1978). Table 3. represents the variation in water and saliva sorption value of dental restoratives after 35 days.

As per the results of this study, Artificial saliva have more prominent effect on the sorption and solubility characteristics of DRCs as compared to distilled water. DRCs-12M shows lower solubility in water i.e., 1.60563 $\mu\text{g}/\text{mm}^3$ whereas in saliva it is 1.90879 $\mu\text{g}/\text{mm}^3$ and DRCs with 4 wt.% of n- HAPs filler shows maximum solubility (3.69765 $\mu\text{g}/\text{mm}^3$ in distilled water and 4.13859 $\mu\text{g}/\text{mm}^3$ in artificial saliva) in water as well as artificial saliva as shown in Figure 1 and Figure 2. Increased solubility of dental restoratives is related to the leaching of free unreacted monomers, fillers and silane additives (Landuyt, et al., 2011; Söderholm, et al., 2000).

Table 3. Variation in water and saliva sorption value of dental restoratives after 35 days

S. No.	Composite Series	Water Solubility ($\mu\text{g}/\text{mm}^3$)	Water Solubility (%)	Saliva Solubility ($\mu\text{g}/\text{mm}^3$)	Saliva Solubility (%)
1.	DRC-0	3.40904	0.28985	3.97724	0.67633
2.	DRC-4M	3.69765	0.3027	4.13859	1.23296
3.	DRC-8M	2.72645	0.18948	2.42825	0.54801
4.	DRC-12M	1.60563	0.09093	1.90879	0.17249
5.	DRC-4A	3.84024	0.33542	4.28576	1.45807
6.	DRC-8A	2.91679	0.12547	2.72289	0.61459
7.	DRC-12A	1.8357	0.08354	1.96454	0.14825

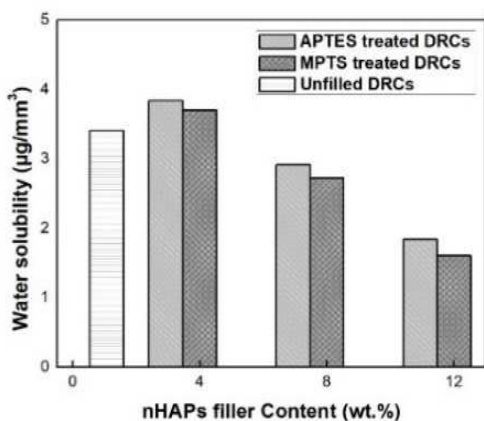


Figure 1. Effect of APTES/MPTS treated n-HAPs fillers on water solubility of DRCs (after 35 days)

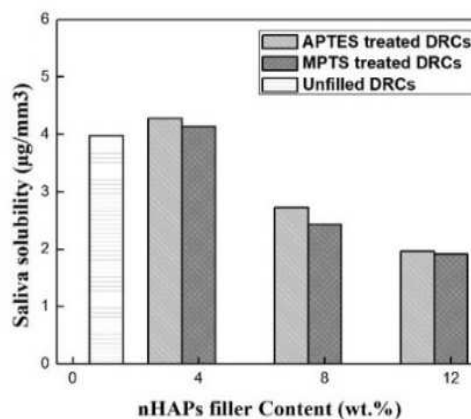


Figure 2. Effect of APTES/MPTS treated n-HAPs fillers on saliva solubility of DRCs (after 35 days)

Also, the leaching of unreacted monomers is affected by its hydrophilicity and mobility with HEMA/TEGDMA i.e., most susceptible monomer to leach in dental restoratives (Alshali, et al., 2015). The sorption value in water (Fig.3 and Fig.4) and artificial saliva (Fig.5 and 6) decreases with increase in the weight fraction of functional n-HAPs in the DRCs as shown in Table 4 and Table 5.

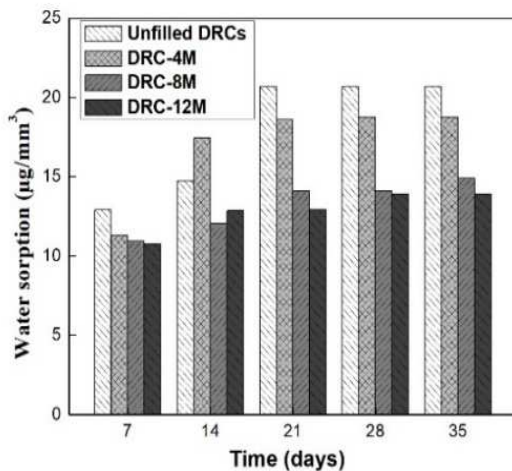
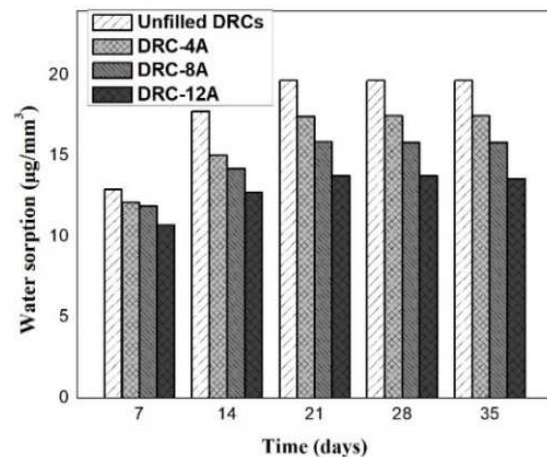
This result is associated to the presence of substantial amount of hydrophilic groups such as TEGDMA and HEMA in the organic matrix part of the DRCs i.e. absorb greater amount of water and artificial saliva in unfilled DRCs (Mese, et al., 2008; Alshali, et al., 2015). The surface functionalization of n-HAPs increases the hydrophobicity of the fillers which in turn provide more resistance to sorption and solubility phenomenon in DRCs.

Table 4. Effect of MPTS/APTES treated fillers on the water sorption behavior of dental restoratives in 35 days

S. No.	Composite Series	Water Sorption ($\mu\text{g}/\text{mm}^3$)				
		7 days	14 days	21 days	28 days	35 days
1.	DRC-0	12.92974	14.7495	20.67652	20.67652	20.67652
2.	DRC-4M	11.3391	17.45634	18.6062	18.7676	18.7676
3.	DRC-8M	10.92956	12.0347	14.10482	14.11847	14.8965
4.	DRC-12M	10.7488	12.8637	12.8965	13.8965	13.8965
5.	DRC-4A	12.11942	15.01457	17.4562	17.4735	17.4735
6.	DRC-8A	11.8731	14.2231	15.8397	15.8252	15.8252
7.	DRC-12A	10.6944	12.7495	13.76062	13.76062	13.57482

Table 4. Effect of MPTS/APTES treated fillers on the saliva sorption behavior of dental restoratives in 35 days

S. No.	Composite Series	Saliva Sorption ($\mu\text{g}/\text{mm}^3$)				
		7 days	14 days	21 days	28 days	35 days
1.	DRC-0	16.4393	19.09078	21.39164	21.87337	21.87337
2.	DRC-4M	14.4269	16.97649	17.63942	17.63942	17.63942
3.	DRC-8M	13.8893	13.81119	13.81119	14.00375	14.00375
4.	DRC-12M	11.02242	12.13363	12.96165	12.96165	12.96165
5.	DRC-4A	15.87906	17.97649	18.96156	18.96156	18.96156
6.	DRC-8A	14.93363	15.5629	16.34268	16.83076	16.83076
7.	DRC-12A	12.08357	13.7925	13.58499	13.58499	13.58499

**Figure 3.** Effect of MPTS treated n-HAPs fillers on water sorption behavior of DRCs**Figure 4.** Effect of APTES treated n-HAPs fillers on water sorption behavior of DRCs

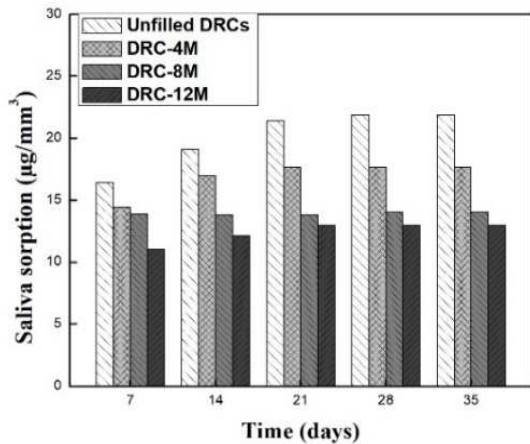


Figure 5. Effect of MPTS treated n-HAPs fillers on saliva sorption behavior of DRCs

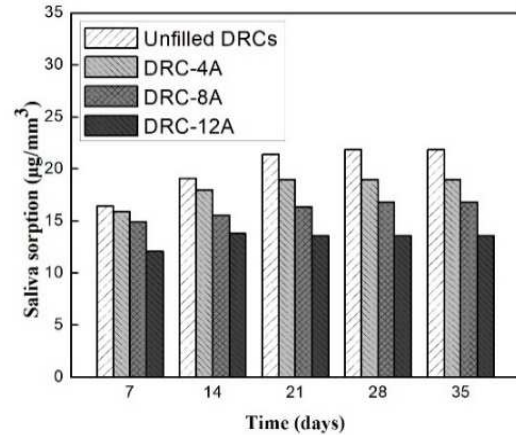


Figure 6. Effect of APTES treated n-HAPs fillers on saliva sorption behavior of DRCs

Table 4 and Table 5 reveal that greater water and saliva sorption occurred within the initial days of immersion and it gets saturated within the 3-4 weeks of immersion. This phenomenon is associated to the polymerization process that gets completed over a period, also the void entrapped into the DRCs adversely affects the polymerization efficiency of the materials and increase its sorption and solubility. Many studies have reported variation in the sorption and solubility depending on its physiochemical properties of materials even though the main constituents of the materials are same (Alshali, et al., 2015; Knoblock, et al., 2000). After 35 days, slightly increased water sorption was detected in MPTS treated n- HAPs filled DRCs (DRCs-M) than that of APTES treated n-HAPs filled DRCs (DRCs-A), but this difference was decreases with increasing the filler contents (i.e., 13.8965 $\mu\text{g}/\text{mm}^3$ for DRCs-12M and 13.5748 $\mu\text{g}/\text{mm}^3$ for DRCs- 12A) as shown in Fig. 3 and Fig. 4 respectively. However, polymers are characterized for variable degree of solubility and sorption depending on the existence of hydroxyl groups (i.e., forms hydrogen bond with water), their micro-polarity of molecules, degree of polymerization of matrix, nature and volume percent of fillers, presence of water absorbing monomers in matrix (i.e., HEMA and TEGDMA) (Eisenburger, et al., 2003).

4. Conclusions

Sorption and solubility of dental restorative composites in a specific medium are material-sensitive properties and significantly influenced by the volume fraction of fillers, monomer conversion and properties of the polymer matrix. After the continuous observation of 35 days, it has been found that distilled water and artificial saliva medium are comparable as storing medium in terms of sorption and solubility. DRCs are more susceptible to artificial saliva medium for moisture uptake and solubility as compared todistilled water due to additional constituent of the artificial saliva medium. Incorporation of silanized n-HAPs fillers, reduces the solubility and moisture uptake phenomenon due to enhanced hydrophobicity of fillers and at higher filler loading (12 wt.%), these MPTS/APTES treated DRCs are showing improved solubility and sorption value as compared to unfilled DRCs. The result of this study shows that DRCs-12M and DRCs-12A could be considered as promising material in dental restoration, as they performed better in terms of moisture sorption and solubility i.e.,within the acceptable limit of the ISO 4049 standard.

The dimensional stability, structural and chemical integrity are some crucial features that evaluate the durability and clinical success of dental restorations in oral cavity. Depending upon the eating habits of patients these materials are invariably exposed to different medium apart from saliva-distilled water condition such as citreous food and drinks, coke, food slurry etc. The effect of these oral mediums also needs to be considered for the for sorption and solubility study of dental restoratives, that will give the better understanding of actual oral conditions.

Nomenclature

- Bis-GMA Bisphenol A glycidyl methacrylate
- HEMA 2-hydroxyethyl-methacrylate
- TEGDMA Triethylene glycol-dimethacrylate
- CQ Camphorquinone
- DMAEMA di-methylamino ethyl methacrylate,
- MPTS(3-methacryloxypropyl) trimethoxy silane
- APTES (3-aminopropyl) triethoxysilane

n-HAPnano-hydroxyapatite
 W_{sp} Water Sorption
 $W_{s|}$ Saliva Sorption

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