Dental Light-Cured Polymer Nanocomposites: A Brief Review of Experimental Results Obtained from the Study of Some Physicochemical Properties

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Abstract: This article is a brief review of the experimental results obtained from the study of some physicochemical properties of five dental commercial light-cured nanocomposites: Filtek Supreme XT Body (*FSB*) and Filtek Supreme XT Translucent (*FST*), both characterized as nanofill composites, and Grandio (*GR*), Tetric EvoCeram (*TEC*) and Protofill-nano (*PR*) characterized as nanohybrid composites. The properties presented are: volumetric shrinkage due to polymerization, sorption, solubility and volume increase after sorption of composites in water or artificial saliva *SAGF*® medium for 30 days, flexural strength and modulus after immersion in water or artificial saliva for one or 30 days and finally the dynamic mechanical properties (storage modulus E´, loss modulus E´´, tanδ=E´´/E´ and T_g) determined 1h after light-curing or after immersion in an oral/food simulating liquid (water, artificial saliva or ethanol/water solution 75%vol) for 1 month. It was found via physicochemical properties that the most corrosive solvent is EtOH which heavily affects the polymeric matrix, while water and saliva are smoother media. Moreover, the more rigid and concrete polymeric chains may sustain higher load during mechanical tests, so that the composites with stronger networks shall demonstrate resistance.

Keywords: Dental nanocomposites, food simulating liquids, volumetric shrinkage, sorption, solubility, volume increase, flexural properties, viscoelastic properties.

INTRODUCTION

Nanocomposite materials, synthesized by inorganic fillers of the nanoscale and dimethacrylate esters forming a polymer network matrix, find wide applications in contemporary dental restorations. Strong onlays are demanded for posterior applications, while beautiful ones for anterior teeth; their combination produces the ideal product! The structural characteristics and physico-chemical properties of some current commercial light-curing dental nanocomposites are included in this piece of work.

Trials and efforts were carried out to induce, the well-known, poly(methyl methacrylate) (PMMA) in biomedical applications, such as dental restorations, since the end of World War II. Its glassy form incommoded the work, but fifteen years later, a larger methacrylate ester bearing two vinyl bonds, that is Bis-GMA (Bowen's ester) (Figure **1**), proved to be more proper. Based on that, other similar molecules were synthesized as well. Also, mechanical enhancement was achieved through inorganic fillers addition, their variety is noticeable. The decades of 1970-80 produced several types of composites, classified by the size of their fillers' particles, from macro- to micro- and mini-scale or hybrids.

The curing techniques, of the methacrylates originally and the composites then, involved heating till 1960s, cold (so called self-) polymerization for some years later and photocuring from 1975 and on. UV- and Vis-light curing today cover polymers' requirements.

Little after 2000, nanotechnology and advanced materials entered this field too, diminishing the particles' size to 100nm or less. Other detailed modifications in the existing ingredients improve the overall correspondence too. Nanocomposites today is the spearhead of novel materials based on polymeric matrix [1].

DENTAL LIGHT-CURED POLYMER NANOCOMPOSITES

Composition

Light-cured polymer nanocomposites consist of two major phases: the organic polymeric matrix and the inorganic filler. Initiation system, coupling agent and other additives are vital for the efficiency of the material as well. Bisphenol A-glycidyl methacrylate (Bis-GMA) or bisphenol A-ethoxylated methacrylate (Bis-EMA) with their aromatic rigid structures can form the stable and strong polymer network matrix needed. The opening of two vinyl bonds for each monomer leads to crosslinking and three-dimensional network. Incomplete polymerization and low degree of conversion are faced via addition of flexible aliphatic

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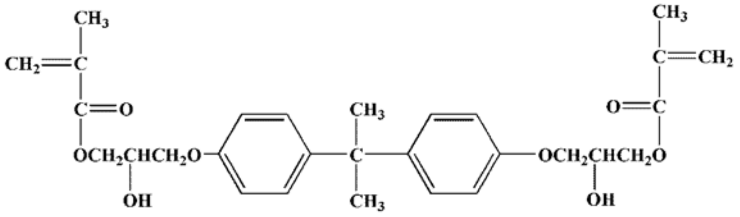
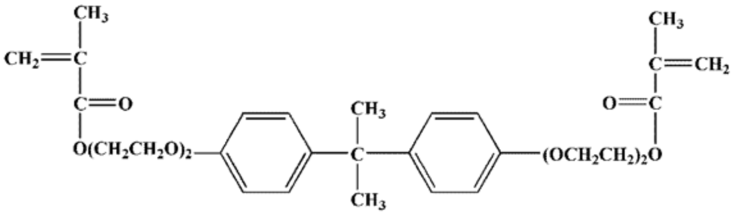
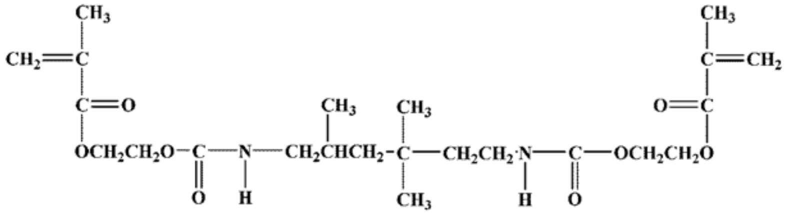


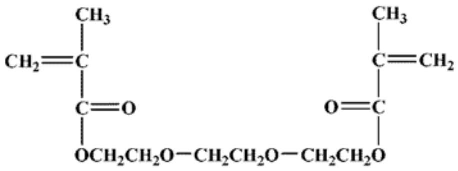
Bis-GMA



Bis-EMA



UDMA



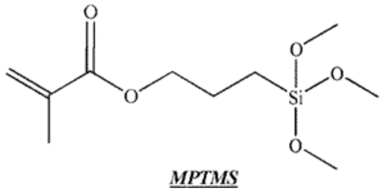
TEGDMA

Figure 1: Structure of dimethacrylates frequently used in dental polymer composites.

co-monomers such as triethylene urethane dimethacrylate (UDMA) or triethylene glycol dimethacrylate (TEGDMA) (Figure **1**) [2]. The choice and the exact portion of co-monomers affect strongly most of the properties in short and long term.

Quartz, silica, zirconia, silicates, oxides often compose the inorganic part of the material. Newer substances involved are ceramics, organically modified ceramics or prepolymerized resins. All those fulfill a number of required properties, absence of toxicity, colour stability and inert chemical character among them. It has been rather established to sort the types of composites in market by the size or the shape of the fillers' particles. The reinforcement of mechanical properties is significant for posterior teeth, while the smoothness for anterior applications [3]. Nanofillers have conquered the manufacturers' preferences due to their exceptional benefits in both cases.

Organosilane coupling agents are compounds added in minor quantities in order to unite the matrix to the fillers' grains effectively through steady covalent bonds and not only simple contact or interactions. Cracking and agglomeration must be avoided for a long-time applied product. Silanes as coupling agents bear both groups: an acrylic for chain attachment through polymerization and a Si–OH for creation of Si–O–Si bridges on grains' surfaces. 3-Methacryloxy-



MPTMS

Figure 2: Chemical structure of 3-Methacryloxypropyl-trimethoxysilane (MPTMS) coupling agent.

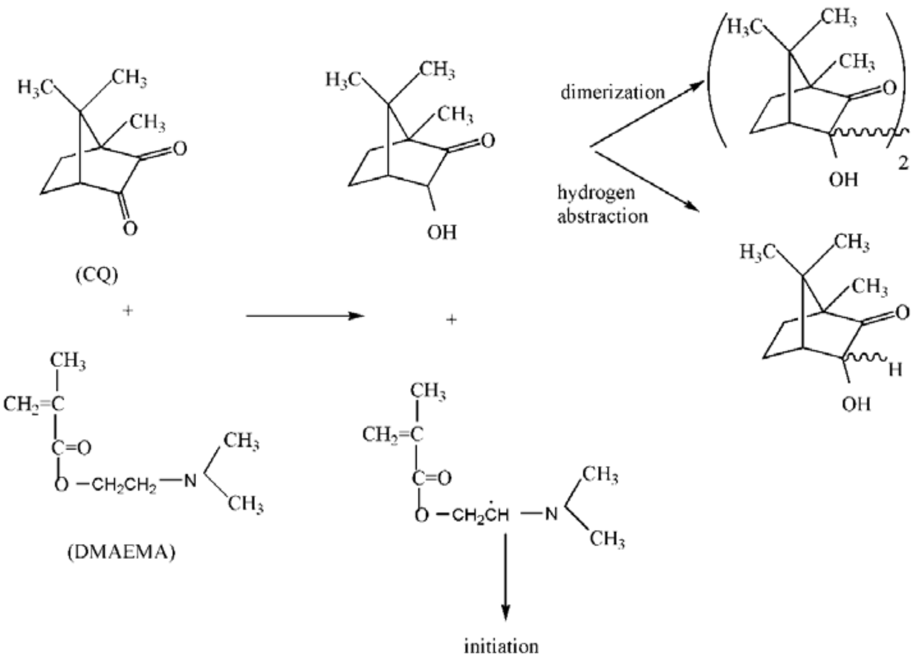


Figure 3: Mechanism of photoinitiation of polymerization by camphorquinone/amine system.

propyltrimethoxysilane (MPTMS) is the most commonly used silane (Figure **2**) [4].

Camphorquinone, as initiator, is excited by light and along with a tertiary amine [e.g. 2-(N,N dimethylamino) ethylmethacrylate, DMAEMA] produces the radicals needed for initiating the chain polymerization reactions (Figure **3**). This system works great at room temperature (absence of heating) and in short time (convenient in practice).

Colour pigments, UV-stabilizers, inhibitors and YbF_3 are some of the rest substances added in the composites.

Photopolymerization (Light-Curing)

The polymerization of dimethacrylates follows the free-radical mechanism and is mainly initiated by light. Camphorquinone is a yellow solid substance which absorbs at 468-470nm. When blue light (complementary colour) radiates a sample radicals are instantly produced, the first monomers are struck, then the neighboring ones etc. The stages of initiation, propagation and termination of polymerization are carried out as known. While polymerization proceeds the material becomes stiff and rigid so the polymerization rate decreases. The main concern, apart from insufficient conversion, is polymerization shrinkage, which can create problems like cracks, irregularities or even abruption with possible clinical dangers [5,6].

The apparatus activating this type of polymerizations includes halogen-tungsten devices, LED, laser or plasma. The operator should make sure that the emission wavelengths of the particular device cover sufficiently the absorption wavelengths of the initiation system contained.

Oral/Food-Simulating Liquids

The restorative materials after been placed spent their lifetime in mouth, coming in contact with food and drinks, saliva and facing the personal hygiene of each patient. This environment changes several times a day (during meals, after those or at night), thus chemical/ biochemical conditions change too affecting their wearing. In order to simulate satisfactorily the mouth conditions that dental materials face, Food & Drug Administration has created a list of solvents or solutions which can be utilized as oral/food-simulating liquids (FSLs) (Table **1**) [7]. Specimens aged or worn

Table 1: Food/Oral-Simulators Matching

Food	FSL
Water, aqueous solutions	H_2O
Alcohol, refreshments, soft drinks, sweets	EtOH/EtOH in H_2O
Oils, fats, meat	Hexane/Heptane
Salty, sauces, dressings	NaCl in H_2O
Human saliva	Artificial saliva

into those liquids would give us a picture on how corrosive and intensive a specific environment is. The time keeping the specimens into the liquids may seem weird and long compared to the fleeting time food stays in mouth, but is a sign of the endurance to the damage that will be shown anyway in long terms.

Table 2: *SAGF® Medium* Ingredients[9]

Compound	Concentration (mg/L)
NaCl	125.6
KCl	963.9
NH_4Cl	178.0
KSCN	189.2
$NaHCO_3$	630.8
NH_2CONH_2	200.0
KH_2PO_4	654.5
$CaCl_2 \cdot 2H_2O$	227.8
$Na_2SO_4 \cdot 10H_2O$	763.2

Water is without doubt the first solvent to be studied because of its importance and presence everywhere. The 75% vol. $EtOH/H_2O$ on the other hand not only simulates the organic environment and alcohol particularly, but presents the same solubility coefficient to Bis-GMA [8]. That is, this solution affects deeply the polymer and subsequently deserves to be investigated. As for the artificial saliva, the reasons for study are obvious. Human saliva is a complex system including water, electrolytes, amino-acids, hormones, enzymes and mucus. Its exact composition and pH values change from time to time during the day, due to which is difficult to be accurately simulated. Literature includes several recipes of artificial saliva the one chosen for our study is shown below in Table **2**. Crucial topics are the choice of organic compounds, the pH setting, presence of CO_2, ionic strength, conductivity, Ca^{2+} concentration etc. [9].

EXPERIMETAL RESULTS OBTAINED FROM THE STUDY OF SOME PHISICOSHEMICAL PROPERTIES OF THE COMPOSITES

Five current commercial light-cured dental nanocomposites were selected and studied recently in our Laboratory. The characteristics of these composites are shown in Table **3**. The aim of the study was to notice the differences in some physicochemical properties and try to attribute them to the differences in the amount and/or chemical composition of polymer matrix or fillers amount and type. For each procedure followed, samples were prepared after photocuring using a dental light apparatus (XL 3000 from 3M Company, St. Paul, MN, USA) and drying onto a desiccant for removing surfacing moisture. The shape and the size of each tester-sample depended on the experimental requirements.

Table 3: Information Accompanying the Composites Given by the Manufacturers

Brand name	Company	Type	Organic matrix (monomers)	Reinforcing filler
Filtek Supreme XT Body **FSB**	3M ESPE	Nanofilled	Bis-GMA, Bis-EMA UDMA, TEGDMA 21.5 %wt	SiO_2: 5-20 nm nanoparticles (8%wt) Zirconia/silica nanoclusters 0.6-1.4 μm (71%wt) 79 %wt (total filler)
Filtek Supreme XT Translucent **FST**	3M ESPE	Nanofilled	Bis-GMA, Bis-EMA UDMA, TEGDMA 27.5 %wt	SiO_2: 75 nm nanoparticles (40% wt) SiO_2 : 0.6-1.4 μm nanoclusters 30%wt (70 %wt total filler)
Grandio **GR**	Voco	Nanohybrid	Bis-GMA, TEGDMA *13 %wt*	SiO_2: 20-60 nm Al, B, Ba silicates: 0.1-2.5μm mean 1.0 μm 87%wt (total filler)
Tetric EvoCeram **TEC**	Ivoclar Vivadent	Nanohybrid	Bis-GMA, UDMA *17-18 %wt*	SiO_2, Ba salts, oxides, prepolymerized resin 0.04-3.0 μm, mean 0.55 μm 82-83%wt (total filler)
Protofill-nano **PR**	a Greek product	Nanohybrid	Bis-GMA, UDMA, TEGDMA *18 %wt*	nanoparticles: 20 nm Sr, Al, B silicates: 0.6 μm 81.9%wt (total filler)

Volumetric Shrinkage

The polymerization of methacrylate-based composites is subject to contraction because the distance between the monomer molecules before polymerization is lower than the distance of the monomer units in the polymer chain. Polymerization shrinkage is one of the dental clinicians' main concerns when placing direct polymer based composite restorations, because it may lead to poor marginal seal, marginal staining and recurrent caries [10,11]. The volumetric shrinkage (%) was measured based on Archimedes' Principle as described in literature [12,13]. The results obtained are presented in Figure **4**. The volumetric shrinkage values follow the order: Grandio (*GR*) <Tetric EvoCeram (*TEC*) < Protofil-nano (*PR*) Filtek Supreme XT Body (*FSB*) < Filtek Supreme XT Translucent (*FST*) [14]. This order seems to depend on the total amount of filler of composites. *GR* contains the highest amount of filler (87%wt) and *FST* the lowest (72%wt) (Table **3**). Polymer composites with high filler content have higher Young's modulus of elasticity, which will reduce volumetric construction (because of the higher filler content relatively to the lower polymer matrix content). However, *PR* showed shrinkage than *TEC*, although both contain about the same of filler (≈ 82%wt). This may be attributed to the different chemistry of the polymer matrix. The matrix of *PR* consists of Bis-GMA, UDMA and TEGDMA, while that of *TEC* consists of Bis-GMA and UDMA. It is known that the polymerization shrinkage of composites depends on the degree of conversion of matrix monomers during polymerization; the greater the degree of polymerization the greater the shrinkage. It is also known that TEGDMA shows a much greater degree of conversion than both Bis-GMA and UDMA [15].

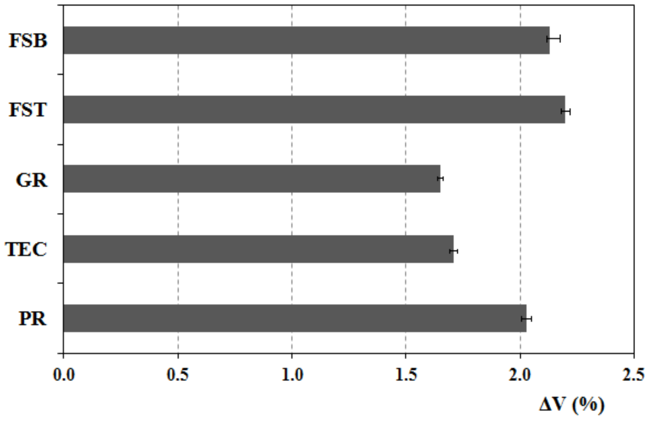


Figure 4: The volumetric shrinkage (%) of the studied composites after 60s-polymerization.

Sorption of Water or Artificial Saliva-Solubility-Volumetric Change

After polymerization, composite discs (15mm x 1mm) were immersed in water or artificial saliva SAGF® for 30 days. Sorption and solubility tests were carried out according to the method described in ANSI/ADA Specification No. 27-1993 (ISO 4049) and in Ref. [16]. The results obtained are presented in Table **4** (sorption) and Table **5** (solubility and volume increase).

Table 4: Sorption of Water or Saliva SAGF® at 37°C by Composites after Immersion for 30 Days

Composite	% Sorption	
	H_2O	SAGF®
FSB	1.59±0.01	1.58±0.02
FST	1.66±0.04	1.67±0.03
GR	0.68±0.01	0.63±0.01
TEC	1.10±0.02	1.11±0.02
PR	1.30±0.02	1.29±0.02

means±st. dev. of n=4 replicates

Table 5: Solubility and Volume Increase of Composites after Immersion for 30 Days in Water or Saliva SAGF® at 37°C

Composite	% Solubility		% Volume Increase	
	H_2O	SAGF®	H_2O	SAGF®
FSB	0.25±0.05	0.28±0.01	0.82±0.17	0.75±0.74
FST	0.22±0.02	0.22±0.04	1.00±0.12	0.92±0.71
GR	0.09±0.02	0.06±0.02	0.00±0.00	-0.22±0.67
TEC	0.09±0.02	0.09±0.01	0.41±0.62	1.26±0.40
PR	0.28±0.03	0.36±0.04	1.12±0.64	0.71±0.38

means±st. dev. of n=4 replicates

Water or artificial saliva uptake in the polymer matrix of composites causes generally *i)* extraction of the unreacted monomers resulting in shrinkage, loss of weight and reduction in mechanical properties and *ii)* swelling of the composite and an increase in weight. The solvent diffuses into the polymer matrix and separates the polymer chains creating expansion. However, since the polymer network contain microvoids created during polymerization, a part of the solvent is accommodated in these voids without causing a change in volume [17,18]. Thus the dimensional change of a polymer composite in a solvent depends on the chemical structure of the polymer matrix. The amount of water which was absorbed follows the order: *GR<TEC<PR<FSB<FST* (Table **4**). This order indicates that water sorption

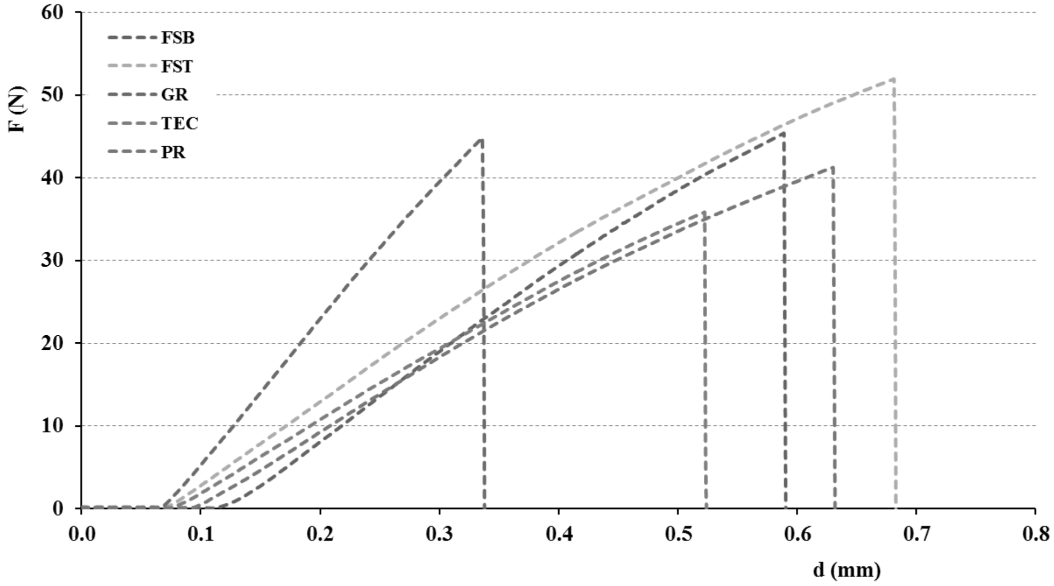


Figure 5: Flexural strength of composites after aging in water for 30d.

Table 6: Flexural Strength (σ) of Composites after Immersion in Water or Saliva SAGF® 37°C for 1 Day or 30 Days

Composite	σ (MPa) after 1 day		σ (MPa) after 1 month	
	H_2O	SAGF®	H_2O	SAGF®
FSB	144.36±9.84	147.93±5.65	95.75±4.87	86.36±8.20
FST	103.69±11.14	127.19±26.45	94.23±11.19	80.46±12.81
GR	116.37±3.70	112.24±0.86	116.13±5.28	101.49±3.91
TEC	98.88±5.94	105.39±3.20	79.49±8.11	76.94±6.55
PR	107.47±7.47	115.97±10.39	98.04±2.69	79.91±6.08

means±st. dev. of n=5 replicates

depends on the content of polymer matrix. However *PR*, which has the same matrix content with *TEC* showed statistically higher water sorption. *PR* contains TEGDMA, which is known to be more hydrophilic than Bis-GMA and UDMA [19]. The *GR* and *TEC* with lower content of polymer matrix showed also lower solubility than that of *PR*, *FSB* and *FST* (Table **5**). The amount of absorbed water, the solubility and the volume increase is not statistically different from the corresponding quantities observed in the case of artificial saliva.

Static Mechanical Properties

Mechanical properties cover an important part of samples' characterization in total, since they do come in touch with tools and instruments that cause damage during their shaping, polishing or other dental works. Later into mouth, daily brushing, eating harsh food etc. affect their endurance and durability. 3-point-bending is an easily executed static test measuring the flexural strength and modulus of a tester sample of specific geometry. Solvents' wearing is certainly aggravating for the mechanical properties, but a strong matrix and great quantities of filler may protect the overall behavior. Water or SAGF® medium were used for aging the samples for one day or one month before mechanical tests according to the method described in ANSI/ADA Specification No. 27-1993 or ISO 4049 and in Ref. [14]. Curves such as shown in Figure **5** were obtained.

Data processing through certain equations leads us to flexural strength σ (Table **6**) and flexural modulus *E* (Table **7**).

The results presented in Tables 6 indicate that the flexure strength depends not only on the filler content but also on the filler chemistry [20,21]. After immersion in water for 30 days the flexural strength of *TEC* remained constant, while that of all other composites decreased. The values of flexural strength after immersion in water or artificial saliva for one day are comparable. When the composites remained in water or saliva for 30 days the saliva had a stronger effect on

Table 7: Flexural Modulus (*E*) of Composites after Immersion in Water or Saliva SAGF® 37°C for 1 Day or 30 Days

Composite	*E* (GPa) after 1 day		*E* (GPa) after 1 month	
	H_2O	SAGF®	H_2O	SAGF®
FSB	10.14±0.21	10.49±0.20	9.21±0.26	9.75±0.24
FST	8.42±0.22	8.54±0.22	7.58±0.12	8.03±0.22
GR	14.81±0.07	14.92±0.32	14.44±0.76	14.91±0.32
TEC	7.72±0.20	8.03±0.14	6.94±0.33	7.33±0.23
PR	8.36±0.24	8.76±0.10	8.24±0.33	8.46±0.17

means±st. dev. of n=5 replicates

flexural strength for *GR* and *PR* and no significant difference was observed for the other composites. Flexural modulus values in water or saliva did not show any significant difference for one day or 30 days (Table **7**).

Dynamic Mechanical Analysis (DMA)

DMA is suggested as a valuable tool for characterization of viscoelasticity of dental composites giving a greater insight into their mechanical behavior [22-24]. Dynamic analysis is preferred than the static for composite materials because they go through a repeating procedure during their lifetime, such as mastication. The sinusoidal oscillation of *f*=1Hz applied in DMA resembles the movement of the jaws during eating. The viscoelastic materials like the polymer composites, present two moduli; the storage or elasticity modulus (E´) which is a measure of the elastic behaviour of the composite and the loss or viscous modulus (E´´) which is a measure of the viscous behaviour. The ratio E´´/E´=tan*δ* called tangent delta is a dimensional parameter which is a measure of the energy lost to energy stored in a cyclic deformation in DMA. The peak of the tan*δ* curve corresponds to the glass transition temperature (T_g) of composite (Figure **6**). In fact, the DMA is the recommended technique for measuring T_g of crosslinked polymers. The half-peak width (ΔT) and maximum height of the tan*δ* curve can give information about the heterogeneity of composite [25].

Composites remained in water, saliva SAGF® or EtOH solution (75%vol) for one day or week or month or 3-months before DMA. The results obtained from DMA of nanofilled composites *FSB* and *FST* were published in Ref. [25], while those of nanohybrid *GR*, *TEC* and *PR* in Ref. [26]. Some of these results are shown in Table **8**. The most interesting result is that all composites without aging in a liquid showed two T_g (two tan*δ* peaks). The first one, at about 40-43°C, is attributed to the composite after light-curing and the second one, at much higher temperatures, to the composite post-cured during the DMA measurement (heating from 25°C to 185 °C with a heating rate of 2 °C/min). The two tan*δ* peaks for the unworn specimen

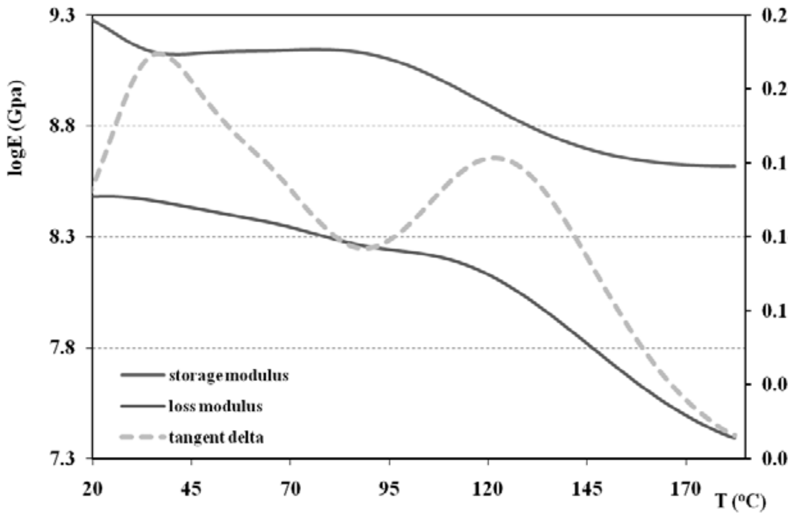


Figure 6: A typical diagram obtained from DMTA.

Table 8: Viscoelastic Properties of Composites at 37°C and T_g after Immersion in a Solvent for a Month

Composite	E´ (GPa)	E´´ (GPa)	tan*δ* (x10^3)	T_g (°C)
FSB unworn	5.46±2.12	0.55±0.36	101±4	43.5±4.0/104.7±1.4
H_2O	5.53±1.39	0.56±0.17	102±6	75.0±2.8
SAGF®	5.48±0.23	0.58±0.07	105±1	76.4±9.1
EtOH solution	3.18±1.24	0.40±0.25	126±8	61.5±4.6
FST unworn	6.08±0.53	0.76±0.01	125±1	43.3±1.4/100.6±1.0
H_2O	5.19±0.19	0.54±0.09	102±4	71.9±1.9
SAGF®	5.90±0.75	0.64±0.07	108±6	72.9±1.1
EtOH solution	4.46±0.16	0.62±0.10	139±8	64.0±1.3
GR unworn	10.99±2.78	0.95±0.15	92±3	40.5±1.5/137.0±4.9
H_2O	9.24±1.22	0.86±0.05	93±5	127.7±1.9
SAGF®	7.66±2.70	0.61±0.14	80±7	125.4±1.2
EtOH solution	7.88±1.70	0.66±0.23	82±5	110.4±2.2
TEC unworn	6.30±0.32	0.68±0.06	110±8	40.1±2.1/113.9±9.6
H_2O	5.43±0.29	0.62±0.03	114±5	81.4±0.6
SAGF®	5.81±0.74	0.56±0.07	97±9	82.7±1.3
EtOH solution	2.37±0.41	0.45±0.07	190±3	64.6±0.8
PR unworn	5.31±0.07	0.86±0.01	161±1	41.7±0.3/134.9±1.6
H_2O	5.48±0.12	0.59±0.04	104±8	94.4±0.8
SAGF®	5.83±1.40	0.52±0.09	91±8	102.4±1.8
EtOH solution	3.78±0.16	0.51±0.02	134±8	77.3±1.7

means±st. dev. of n=3 replicates

are replaced by one wide peak after immersion in a solvent for a certain time (Figure **7**).

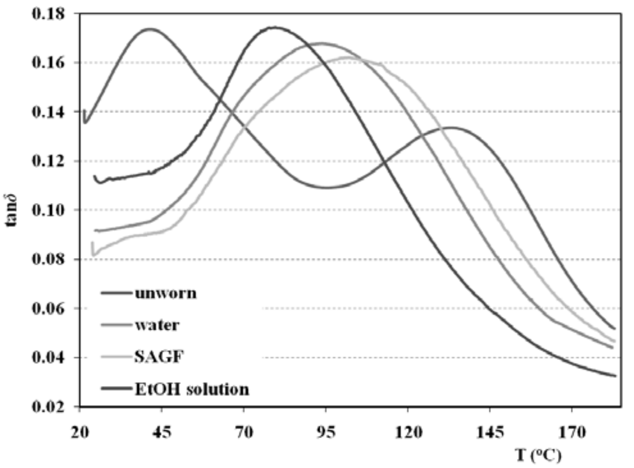


Figure 7: Comparison of tan*δ* of PR composite after immersion in various media for a month.

Using the width of the tan*δ* peak it can be observed that the glass transition temperature region spreads over a wide temperature range. This extended transition region is a result of high degree of structural heterogeneity of sample. The broader tan*δ* peak implies a more heterogeneous network with a wide distribution of relaxation times. Qualitatively this structural heterogeneity is observed by measuring the peak width at half its height of the tan*δ* curve (Figure **8**). It is observed that immersion in EtOH solution (75% vol) results in narrower tan*δ* peaks due to degradation of polymer network chains.

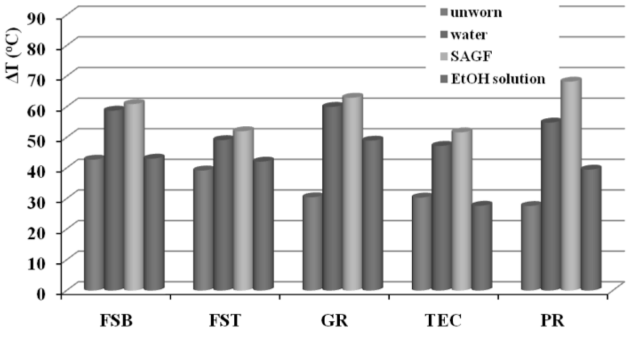


Figure 8: Tan*δ* half-peak width over FSLs after 30-days time for each product.

Storage of composites in water or saliva SAGF® caused a significant post curing and plasticization (reduction of T_g). EtOH solution (75% vol) had a stronger effect than water or saliva SAGF® due to its organophilic nature. It caused post curing, plasticization and most probably degradation of the bond filler- silane coupling agent and/or of bonds of polymeric network.

CONCLUSION

The properties and overall behaviour of a dental nanocomposite is a result of its components; a tough polymeric network enforced with rigid monomer units favours the mechanical properties and endurance of the material but deteriorates the shrinkage and sorption/desorption characteristics. Volume shrinkage of the polymer matrixes must remain in low values, sorption of solvents/solutions percentages as well. In that way desorption and solubility values hold at acceptable levels. Aging and post-curing phenomena are observed through mechanical tests too. T_g values are main findings for the observation, justification and forecast of materials' properties and behavior.

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