Research Article

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Dynamic mechanical behavior of nano-ZnO reinforced dental composite

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Abstract: In the present work, Bisphenol-A Glycidyl Methacrylate / Triethylene Glycol Dimethylacrylate based dental composites filled with 0-30 wt.% silane treated nano-ZnO were fabricated and tested for their dynamic mechanical properties. Samples were kept in each of three different mediums such as cold drink, distilled water and saliva for 7 days. The dynamic mechanical properties such as storage modulus, loss modulus and Tan delta were evaluated and compared for each composite under different conditions. The finding of results indicated that on adding 30 wt.% nano-ZnO, the storage modulus was increased by 109% in case of post cured, 120% in case of cold drink, 125% in case of artificial saliva but decreased by 70% in case of distilled water. The loss modulus was increased by 175% in case of post cured, 30% in case of cold drink, 50% in case of artificial saliva but decreased by 50% in case of distilled water. Further, minimum value of storage modulus was reported in case of distilled water medium followed by cold drink and then artificial saliva. Also, cold drink seems to be better medium than distilled water in terms of dynamic mechanical properties of dental composite.

Keywords: dental composite, nano-ZnO, dynamic mechanical analysis

Graphical abstract: Variation of storage modulus of sample kept in artificial saliva for 7 days

1. Addition of 30 wt.% nano-ZnO increased the storage modulus by 109% in case of post cured, 120% in case of cold drink, 125% in case of artificial saliva but decreased by 70% in case of distilled water.
2. Addition of 30 wt.% nano-ZnO increased the loss modulus by 175% in case of post cured, 30% in case of cold drink, 50% in case of artificial saliva but decreased by 50% in case of distilled water.
3. Immersion of sample in each medium led to decrease in storage modulus but increase in Tan delta.
4. Further, minimum value of storage modulus was reported in case of distilled water medium followed by cold drink and then artificial saliva.
5. Cold drink seems to be better medium than distilled water in terms of dynamic mechanical properties of dental composite.

1 Introduction

Nowadays, experiments are being carried out to find a replacement of amalgam as an environmental concern. In this regards, dental composites are developed and widely used for the direct restoration of both anterior and posterior teeth. The major advantages of dental composites over the other available dental materials include better physical properties, mechanical properties, wear resistance and aesthetic look. The resin based composite consists of base

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resin monomers such as BisGMA, BisEMA, TEGDMA, and fillers such as inorganic oxide, silicates, quartz, and colloidal silica. To achieve stronger bonding between resin and fillers, fillers are coated with the coupling agent. The better adhesion between filler and resin results in good mechanical properties in dental composites. Kumar *et al.* developed and assessed physical, mechanical, thermo-mechanical properties of dental composite materials by modifying resin matrix, filler content/size, silane coupling agent, photo-polymerization system and light curing technique [1, 2]. In general, many types of filler such as boron carbide, silica, nanoalumina and silanes have been tested to improve the mechanical properties of composite materials [3–5]. It has been proposed that increase in filler content, decrease in filler size play the major role in the improvement of dental composites [6]. Also, the surface modification of filler improved the performance of composite material [7]. However, these studies of dental composites have been mainly based on finding static mechanical properties of dental composite. Apart from elastic behavior, dental composite exhibits viscous due to presence of resin matrix. For such viscoelastic materials, static mechanical tests cannot be used to determine viscous response of material. Therefore, Dynamic tests such as dynamic mechanical analysis (DMA) are particularly well suited for viscoelastic materials. Dynamic tests are also used to characterize glass transition of polymeric materials [8]. Dynamic mechanical characterization includes study of storage modulus, viscous modulus and damping factor. The dental composites with low modulus will deform easily under operating condition whereas dental composite with excessive elastic modulus may result in catastrophic fracture of brittle part of tooth structure [9].

Further, it was also reported that the parameters used in dynamic mechanical tests can be used to simulate cyclic masticatory movement of dental composites [10]. The dental composites are usually in contact with oral environment which includes artificial saliva, water, and acidic medium. Therefore, several solvent and medium are used to simulate the environmental conditions for the study of behavior of dental composite. Apart from environmental conditions, light curing intensity, curing temperature and curing technology have influence on the dynamic modulus of elasticity of dental composite [11, 12].

It has been reported that foods and drinks affect the behavior of dental materials. During contact with these medium for long time, some fraction of fillers are separated from resin [13, 14]. As a result, the composite becomes weak and decomposes due to chemical degradation. However, comparative investigation of physical and mechanical properties of dental resin in different work-

ing medium hasn't been presented in literature. Therefore, there is need of clinical and experimental research discussing the dynamic mechanical behavior of dental materials in daily use beverages. Many inorganic oxide fillers such as alumina, silica, and zirconia have long been used to improve the mechanical properties of restorative dental material [15, 16]. Zinc oxide powder has been used in dental composite as antibacterial and opaque reinforcing filler [17]. The viscoelastic study of dental composite in different medium and for different duration is lacking in literature. Application of ZnO to improve dynamic properties of dental composite is also novel research.

In this work, dynamic mechanical behavior of dental composites such as storage modulus, loss modulus and Tan delta of nano-ZnO reinforced dental composites for posterior teeth applications have been studied under three different medium such as cold drink, distilled water, and artificial saliva medium.

2 Experimental details

2.1 Materials used

The conventional base monomers BisGMA (Bisphenol-A Glycidyl Methacrylate) (Esstech Inc, Essington, PA, USA) was taken in a beaker. To add filler in the solution, its viscosity was decreased with the addition of diluent TEGDMA (Triethylene Glycol Dimethylacrylate) (TCI, Tokyo, Japan). 3-methacryloxypropyltrimethoxy-silane, Camphorquinone and Ethyl 4 dimethyl amino benzoate were purchased from TCI, Tokyo, Japan, Spectrochem, Mumbai, India and Sigma Aldrich, Bangalore, India respectively. Nano-ZnO₂ particles (60nm) were procured from Sarthak Sales, Jaipur, India. The physical and chemical properties of Nano-ZnO₂ are presented in Table 1. Artificial saliva (brand Saleva, composition: water, sorbitol, glycerin, gum, sodium saccharin, sodium phosphate, cetylpyridinium chloride) was procured from Global Dent Aids Pvt. Ltd., New Delhi, India. Distilled water and Cold drink (pH 2.6) were procured from local market, Jaipur, India.

2.2 Silane treatment of Nano-ZnO and Fabrication of dental composite

Nano-ZnO₂ particles were treated with 3-methacryloxypropyltrimethoxy-silane to provide better adhesion between resin and filler. Nano-ZnO particles were mixed in the so-

Figure 1: Schematic diagram for the fabrication of dental composite

Table 1: Physical and chemical properties of Nano-ZnO

Particle size	60nm
Density	5500 kg/m ³
Molar Mass	81.40 g/mol
Melting point	1900°C
Boiling point	2200°C

lution of 70/30 wt.% of acetone /water and 15 wt.% of silane relative to filler. Fabrication process of composite has been shown in schematic diagram in Figure 1. Figure 1 showed that initially the resin matrix of BisGMA, TEGDMA, EDMAB and CQ was mixed in the beaker for 2hr and then silane treated fillers were added in the mixture. The complete solution was mixed in the Sonicator for 6h. As per compositions presented in Table 2, four cylindrical specimens ($\varnothing 5.30 \times 6.2 \text{ mm}^2$) were prepared and cured for dynamic mechanical analysis test. Glass slides covered with a mylar sheet (thickness 0.05 mm) were placed on upper and lower surface of the mold to avoid adhesion with the uncured material. LED Light curing unit was used to cure the material up to 40s for each side.

2.3 Transmission Electron Microscopy (TEM):

The morphology of nano-ZnO before and after silane treatment was performed on Transmission Electron Microscope (Tecnai G² 20 S-Twin, USA). The samples were compacted in Cu grid for TEM characterization.

2.4 Dynamic mechanical analysis (DMA)

Viscoelastic materials exhibit both the elastic and viscous properties of the material. Therefore, to study the response of such materials, dynamic mechanical analysis (DMA) is performed. In oral environment, dynamic tests mimic and simulated cyclic mastication process. Therefore, dynamic tests are extremely valuable to predict the experimental and clinical performance of dental composite materials under the cyclic loading during mastication. Dynamic mechanical analysis tests (DMA) were performed in Perkin Elmer Pyris-7 Dynamic Mechanical Analyzer at a frequency of 1 Hz amplitude of 10 μm in the compression mode as per ASTM standard ASTM D5024. Viscoelastic parameters such as storage modulus (E'), loss modulus (E'') and loss tangent ($\tan\delta$) were recorded against temperature in the

Table 2: Designation and composition of composites

	Resin Matrix				Filler Nano-ZnO (% of Resin Matrix)
	Bis –GMA (wt.%)	TEGDMA (wt.%)	DMAEMA (wt.%)	CQ (wt.%)	
DCZnO-0	54	45	0.8	0.2	0
DCZnO -10	54	45	0.8	0.2	10
DCZnO -20	54	45	0.8	0.2	20
DCZnO -30	54	45	0.8	0.2	30

Figure 2: TEM images of nanoZnO filler (a) after silane treatment (b) before silane treatment

range of 25–150°C at a heating rate of 10°C/min. The cylindrical specimen of 4 mm diameter and 6.2 mm height have been fabricated and divided into groups of three samples each.

nanoparticle was covered with the layer of silane. Formation of silane layer improved the adhesion between filler and resin matrix. Better adhesion led to better interfacial force between filler and resin due to formation of siloxane bond [18].

3 Results and discussion

3.1 Transmission electron microscopy analysis of nano-ZnO filled dental composites

TEM analysis was performed by preparing Cu grid of silane treated and untreated nano-ZnO filler. TEM images of untreated and treated fillers were taken at the magnification of 50 nm and depicted in Figure 2(a) and Figure 2(b) respectively. Figure 2(a) showed agglomerated and dispersed nano-ZnO. Further, silane treatment resulted in uniform dispersion of nano-ZnO which can be confirmed from Figure 2(b). It can also be observed from Figure 2(b) that the

3.2 Dynamic mechanical analysis of nano-ZnO reinforced dental composite

The behavior of material under oscillating loading kept under cold drink, distilled water and artificial saliva is studied using DMA test in terms of storage modulus (E'), loss or viscous modulus (E'') and $\tan \delta$ (ratio of loss to storage modulus). The period of immersion under these medium was kept at 7 days. The storage modulus represents the elastic properties of a viscoelastic material. The loss modulus represents viscous properties of a viscoelastic material and related to energy lost during viscous flow. The $\tan \delta$ is used to measure damping properties of ma-

Figure 3: Variation of storage modulus of (a) Post cured sample (b) sample kept in Cold Drink for 7 days (c) sample kept in distilled water for 7 days (d) sample kept in artificial saliva for 7 days

terials. Higher $\tan \delta$ indicates more mobility of molecule inside resin matrix while a low $\tan \delta$ indicates less mobility in the material. Dynamic mechanical analysis of dental composite under different storage conditions and dynamic loading was an attempt of study aging behavior of material.

3.3 Storage modulus (E') of nano-ZnO reinforced dental composite

The storage modulus of *nano-ZnO* filled dental composite after curing was depicted in Figure 3 (a). The storage modulus of *nano-ZnO* filled dental composite kept under cold drink, distilled water and artificial saliva were depicted in, Figure 3 (b), Figure 3 (c) and Figure 3 (d) respectively.

Figure 3 (a) showed the effect of varying temperature, and nano-ZnO content on storage modulus of dental com-

posite after curing. There was general trend of decrease in storage modulus with increase in temperature for all variant of dental composite. It can be seen from Figure 3 (a) that at 20–30°C, the storage modulus was maximum as the composite was made of tightly packed molecule. Afterward, it decreased drastically in the range of 30–100°C indicating glass/rubbery state transition. On further increase in temperature in the rubbery stage, there was no considerable change in storage modulus. Figure 3 (a) also indicated that the storage modulus initially decreased by 25.5% with the increase of filler content up to 10 wt.% and finally increased by 109% with the increase in nano-ZnO content up to 30 wt.%. The increase in storage modulus was attributed to increase in more stiff inorganic particulate fillers. The result is in agreement with the work [19, 20] in which it was reported that increase in stiff filler and better dispersion led to increase in storage modulus.

Figure 3 (b) showed the effect of varying temperature, and nano-ZnO content on storage modulus of dental composite kept in cold drink medium for 7 days. The storage modulus was decreased as compared to that of post cured sample. After 7 days of immersion, DCZnO-0 decreased the storage modulus by 10.10%. The decrease in the storage modulus with the immersion in water was attributed to the plasticization effect. The presence of H-bond with hydroxyl group weakens intermolecular interactions and reduces the inner viscosity [21]. Also in this case, storage modulus was decreased by 52.6% with the increase in filler content up to 20 wt.%, later it increased by 120% with the increase in filler content up to 30 wt.%.

Figure 3 (c) showed the effect of varying temperature, and nano-ZnO content on storage modulus of dental composite kept in distilled water medium for 7 days. In this case, storage modulus increased by 243% with the filler up to 20 wt.%, later it decreased by 70% with the increase of filler content up to 30 wt.%. There was general trend of decrease in storage modulus with increase in temperature in all cases except in the case of DCZnO-20 after 7 days in distilled water storage. In this case, storage modulus initially increased with the increase of temperature up to 80°C but afterward it decreased. The decrease in storage modulus with the increase in temperature was due to increase in molecular mobility inside the polymeric matrix. The increase in storage modulus for DCZnO-20 up to certain temperature may be attributed to the uncured portion of composition which got cured with the increase of temperature.

Figure 3 (d) showed the effect of varying temperature, and nano-ZnO content on storage modulus of dental composite kept in artificial saliva medium for 7 days. It was obvious from the Figure 3 (d) that the Storage modulus was

increased with the increase in filler content and decreased with the increase in temperature. There was maximum decrease in storage modulus when the sample was kept in distilled as compared to the storage modulus of sample when kept in cold drink and artificial saliva medium. Further, storage modulus increased by 125% with the filler up to 30 wt.%. Among all four conditions, there was least storage modulus in case of sample kept in distilled water for 7 days followed by the sample kept in cold drink and then saliva. Further from all four Figure 3(a-d), it was observed that highest storage modulus was shown by composite with 30 wt.% nano-ZnO in post cured, cold drink and saliva, but not shown by this composite in distilled water. It may be due to uncured portion of dental composite reinforced with 30 wt.% nano-ZnO.

3.4 Loss modulus (E'') of nano-ZnO reinforced dental composite

The Loss modulus of *nano-ZnO* filled dental composite was depicted in Figure 4 (a) and the Loss modulus of *nano-ZnO* filled dental composite kept under cold drink, distilled water and artificial saliva were depicted in Figure 4 (b), Figure 4 (c) and Figure 4 (d) respectively. In all cases in Figure 4 (a), Figure 4 (b), Figure 4 (c) and Figure 4 (d), the viscous modulus increased with the increase of temperature. But after reaching glass transition region, loss modulus decreased drastically with the increase of temperature. Figure 4 (a) showed the effect of varying temperature, and nano-ZnO content on Loss modulus of dental composite after curing. Figure 4 (a) indicated that DCZnO-30 exhibited maximum loss modulus as compared to other composite. Loss modulus was increased by 175% when the nano-ZnO was increased up to 30 wt.%. The main reason behind the dynamic test of dental composites in the range of 30–180°C was to find the glass transition temperature. Glass transition temperature (T_g) indicates curing process and physical property of the cured dental composites. The glass transition temperature of dental composite must be higher than the maximum temperature of oral environment to preserve physical and mechanical properties of dental composite. Inadequate curing results in low Glass transition temperature of material. If maximum temperature of oral environment was more than the glass transition temperature of material, the materials can lead to softening and failure of the clinical processes [22].

Figure 4 (b) showed the effect of varying temperature, and nano-ZnO content on loss modulus of dental composite kept in cold drink medium for 7 days. Loss modulus was increased after the immersion in cold drink for

Figure 4: Variation of loss modulus of (a) Post cured sample (b) sample kept in Cold Drink for 7 days (c) sample kept in distilled water for 7 days (d) sample kept in artificial saliva for 7 days

unfilled composite. However, for filled composite, reverse trend was obtained i.e. loss modulus was decreased after immersion in cold drink. Further, increase in filler content initially decreased by 60% for filler content up to 20 wt.% but later it increased by 30% with the increase in filler content up to 30 wt.%. Figure 4 (c) showed the effect of varying temperature, and nano-ZnO content on loss modulus of dental composite kept in distilled water medium for 7 days. It can be seen that loss modulus initially increased by 275% at nano-ZnO up to 20 wt.% but later decreased by 50% at 30 wt.% nano-ZnO. The glass transi-

tion temperature was decreased with the addition of nano-ZnO. Figure 4 (d) showed the effect of varying temperature, and nano-ZnO content on loss modulus of dental composite kept in artificial saliva medium for 7 days. DCZnO-10, DCZnO-20 and DCZnO-30 have shown similar response after reaching glass transition region. With the increase in filler content, it was increased by 50% at nano-ZnO up to 30 wt.%. Among all four conditions, least loss modulus was obtained for the sample kept in distilled water for 7 days as compared to samples kept in cold drink and artificial saliva medium.

Figure 5: Variation of Tan delta of (a) Post cured sample (b) sample kept in Cold Drink for 7 days (c) sample kept in distilled water for 7 days (d) sample kept in artificial saliva for 7 days

3.5 Damping factor ($\tan \delta$) of nano-ZnO reinforced dental composite

The Damping factor ($\tan \delta$) of nano-ZnO filled dental composite after curing was depicted in Figure 5 (a) and the Damping factor ($\tan \delta$) of nano-ZnO filled dental composite kept under cold drink, distilled water and artificial saliva for 7 days were depicted in, Figure 5 (b), Figure 5 (c) and Figure 5 (d) respectively. In all cases in Figure 5 (a), Figure 5 (b), Figure 5 (c) and Figure 5 (d), when tempera-

ture was raised, the loss tangent gradually increased for all series of composites. After reaching its maximum limit in the glass transition region, it decreased drastically.

Figure 5 (a) showed the effect of varying temperature, and nano-ZnO content on Damping factor ($\tan \delta$) of dental composite after curing. Initially, the glass transition temperature decreased by 46.7% with the increase in filler content up to 10 wt.%. Later, the glass transition temperature was increased with the increase in temperature up to 30

wt.%. Further, damping factor was increased with the increase in filler content.

Figure 5 (b) showed the effect of varying temperature, and nano-ZnO content on Damping factor ($\tan \delta$) of dental composite kept in cold drink medium for 7 days. DCZnO-0, DCZnO-20 and DCZnO-30 have shown similar response of Tan Delta (Figure 5 (b)). Figure 5 (c) and Figure 5 (d) showed the effect of varying temperature, and nano-ZnO content on Damping factor ($\tan \delta$) of dental composite kept for 7 days in distilled water and artificial saliva medium respectively. DCZnO-10, DCZnO-20 and DCZnO-30 have shown similar response of Tan Delta (Figure 5 (d)). In all Figure 5(a), Figure 5(b), Figure 5(c) and Figure 5(d), it indicated that keeping samples in cold drink, distilled water, artificial saliva for 7 days widen the peak of the Tan delta versus temperature curves and decreased its amplitude. Therefore, the damping of dental composite was decreased when they were kept in cold drink, distilled water and artificial saliva medium. Therefore, elasticity was improved and material could store more load as compare to dissipation. The widest loss tangent peaks were observed for DCZnO-10, DCZnO-20 and DCZnO-30 kept in distilled water for 7 days (Figure 5(c)).

4 Conclusions

Nano-ZnO₂ particles were treated with 3-methacryloxypropyltrimethoxy-silane. The dental composites reinforced with 0-30 wt.% of nano-ZnO have been fabricated. The dynamic mechanical analysis was performed for the fabricated sample after curing and for the fabricated samples after keeping for 10 days in cold drink, distilled water and artificial saliva. On adding 30 wt.% nano-ZnO, the storage modulus was increased by 109% in case of post cured, 120% in case of cold drink, 125% increase in case of artificial saliva but decreased by 70% in case of distilled water. The loss modulus was increased by 175% in case of post cured, 30% in case of cold drink, 50% in case of artificial saliva but decreased by 50% in case of distilled water. On the other hand, Tan delta was decreased with the increase in filler content. Immersion of sample in each medium led to decrease in storage modulus but increase in Tan delta. Further, minimum value of storage modulus was reported in case of distilled water medium followed by cold drink and then artificial saliva. In the range of 0-20 wt.% nano-ZnO, there was the least decrease in storage modulus in case of distilled water medium followed by artificial saliva and then cold drink. However, at 30 wt.% nano-ZnO, there was maximum decrease in storage modulus in distilled

water medium as compared to post cured, cold drink and saliva medium. It may be due to uncured portion of dental composite reinforced with 30 wt.% nano-ZnO. In terms of dynamic mechanical properties of dental composite, cold drink has shown better choice than distilled water.

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References

- [1] Kumar SR, Patnaik A and Bhat IK. Abrasive wear behavior of silane treated nanoalumina filled dental composite under food slurry and distilled water condition, *Sci Eng Compos Mater.* 2018, 25(3), 541–553.
- [2] Kumar SR, Patnaik A and Bhat IK. Analysis of polymerization shrinkage and thermo-mechanical characterizations of resin-based dental composite reinforced with silane modified nanosilica filler particle. *P I Mech Eng L-J Mat*, 2016, 230, 492–503.
- [3] Abenojar J, Martinez MA, Velasco F, Sanchez VP, Martín-Martínez JM. Effect of boron carbide filler on the curing and mechanical properties of an epoxy resin. *J. Adhes.* 2009, 85, 216–238.
- [4] Abenojar J, Martinez MA, Velasco F, del Real JC. Effect of moisture and temperature on the mechanical properties of an epoxy reinforced with boron carbide. *J. Adhes. Sci. Technol.* 2011, 25, 2445–2460.
- [5] Abenojar J, Martinez MA, Pantoja M, del Real JC. Epoxy composite reinforced with nano and micro SiC particles: curing kinetics and mechanical properties. *J. Adhes.* 2012, 88, 418–434.
- [6] Meena A, Mali HS, Patnaik A, Kumar SR. Comparative investigation on physical, mechanical and thermo-mechanical behavior of nanohydroxyapatite and MTA filled dental composites, *e-Polymers*, 2018, 17(4), 311–319.
- [7] Pantoja M, Diaz-Benito B, Velasco F, Abenojar J, del Real JC. Analysis of hydrolysis process of γ -methacryloxypropyltrimethoxysilane and its influence on the formation of silane coatings on 6063 aluminum alloy. *Appl. Surf. Sci.* 2009, 255, 6386–6390.
- [8] Wilson TW, Turner DT. Characterization of polydimethacrylates and their composites by dynamic mechanical analysis. *J Dent Res*, 1987, 66(5), 1032–1035.
- [9] Jones DW. Dental composite biomaterials. *J Can Dent Assoc*, 1998, 64, 732–734.
- [10] Saber-Sheikh K, Clarke RL, Braden M. Viscoelastic properties of some soft lining materials. I–Effect of temperature. *Biomaterials*. 1999, 20 (9), 817–822.
- [11] Vouvoudi EC, Sideridou ID. Dynamic mechanical properties of dental nanofilled light-cured resin composites: Effect of food-simulating liquids. *J. Mech. Behav. Biomed.* 2012, 10, 87–96.
- [12] Sideridou I, Karabela MM, Spyroudi CS, Dynamic Mechanical Analysis of a Hybrid and a Nanohybrid Light-Cured Dental Resin Composite, *J. Biomater. Sci. Polym. Ed.* 2009, 20(12), 1797–808.

- [13] Harris JS, Jacobsen PH, O'Doherty DM. The effect of curing light intensity and test temperature on the dynamic mechanical properties of two polymer composites. *J. Oral. Rehabil.*, 1999, 26 (8), 635-639.
- [14] Dionysopoulos P, Watts DC. Dynamic mechanical properties of an inlay composite. *J. Dent.*, 1989, 17(3), 140-144.
- [15] Kumar SR, Patnaik A, Bhat IK. Abrasive wear behavior of silane treated nanoalumina filled dental composite under food slurry and distilled water condition, *Sci. Eng. Compos. Mater.* 2018, 25(3), 541–553.
- [16] Meena A, Mali HS, Patnaik A, Kumar SR. Comparative investigation on physical, mechanical and thermo-mechanical behavior of nanohydroxyapatite and MTA filled dental composites, *e-Polymers*, 2018, 17(4), 311–319.
- [17] Aydin SB, Hanley L. Antibacterial activity of dental composites containing zinc oxide nanoparticles. *J. Biomed. Mater. Res. B. Appl. Biomater.* 2010, 94(1), 22-31.
- [18] Gharazi S, Ershad-Langroudi A, Rahimi A. The influence of silica synthesis on the morphology of hydrophilic nanocomposite coating. *Scientia Iranica F.* 2011, 18 (3), 785-789.
- [19] Saude N, Masood SH, Nikzad M, Ibrahim M, Ibrahim. Dynamic mechanical properties of copper-ABS composites for FDM feedstock. *Int. J. Eng. Res. Appl.* 2013, 3, 1257-1263.
- [20] Wei X, Li D, Jiang W, Gu Z, Wang X, Zhang Z, Sun Z. 3D printable graphene composite. *Sci. Rep.* 5, 11181; doi: 10.1038/srep11181 (2015).
- [21] Startsev OV, Krotov AS, Perov BV, Vapirov YM. Interaction of water with polymers under their climatic ageing. In: Milan AIM, editor. *Proc of the 4th European conference of advanced materials and processes—EUROMAT 95*, 1995, 245–254.
- [22] Moraes JCS, Sostena MMDS, Grandini CR. The glass transition temperature in dental composites. In: John, Cuppoletti (Ed.), *Metal, Ceramic and Polymeric Composites for Various Uses*. In-Tech, 2011.