Evaluation of Mechanical Properties of Nylon 6 Nanofiber Reinforced Dental Composite Resins

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Abstract: *Objectives:* The aim of this study was to evaluate mechanical properties of different mass fraction of Nylon 6 (N6) nanofibers reinforced bisphenol A-Glycidyl Methacrylate (Bis-GMA) and tri-(ethylene glycol) dimethacrylate (TEGDMA) based dental composite resins.

Materials and Methods: N6 nanofibers were produced using electrospinning method. The nanofibers were mixed with composite resin and cured. Powder of this mixture was added into the resin matrix at different mass fractions (1%, 2%, 3%). Eight specimens were prepared for each nanofiber reinforced dental composites and neat resin. Three point bending test was applied to specimens. Flexural strength (Fs), flexural modulus (E_Y) and work of fracture (WOF) of groups were found.

Results: Fs results were shown increasing trend going to the highest mass fraction of N6 nanofiber. E_Y result of 2% N6 nanofiber reinforced composite group was significantly increased compared to neat resin. Difference of WOF results between the control and N6 1% nanofiber reinforced composite groups was statistically significant.

Conclusions: Fs and E_Y results of the dental composites could be increased after impregnating relatively small amount of N6 nanofibers. Ratio of surface area to volume could be increased due to inter-molecular hydrogen bonding between the nanofiber and the resin.

Keywords: Dental composite resins, nanofibers, nanofiber reinforced composites, electrospinning.

1. INTRODUCTION

Composite resins, consisting of fillers in a polymer matrix, have been used to restore teeth since their introduction about half-century [1]. Dental composite resins (DCR) are a combination of synthetic polymers, inorganic fillers, initiators, activators and silane coupling agents that bond the reinforcing fillers to the polymer matrix [2].

The composites typically are include three main components: 1) the inorganic fillers, 2) the organic resin matrix, and 3) the silane coupling agents. Although composite resins are widely used in dental practice as a restorative material, improvement of composite resins are necessary. Because, mechanical properties of resins are inadequate and polymerization shrinkage is still high [3, 4]. These three components can be modified, so that composite resins can be reinforced mechanically [2]. However, some authors pointed out addition of the inorganic fillers to the resin may cause to failures instead of fortifying the mechanical properties of the resin. Irregular or angulated shape of inorganic fillers creates excessively high stress concentration points throughout the matrix. This points are developed cracks into the resin. It can cause to cut through or spread around the fillers [5, 6].

During the at least of 10 years, dental nanocomposites have been increased popularity. Thus, too many research projects have been conducted about nanofillers. The researchers hope that increased esthetics, strength and durability of DCRs [2, 3, 5, 7].

Mechanical properties of polymer are improved with nanofibers because of very low filler loadings. The applied load could be transmitted to filler-matrix interface *via* their large interfacial area. At that point, some investigators claimed that the nanofibers have self-tailoring ability to acquire expected mechanical properties [8, 9].

Electrospinning is one of the nanofiber preparation process that is non-mechanical and electrostatic technique to produce from various materials. Polymers, composites and ceramics could be spun using this process [5, 10-12]. In the electrospinning technique, one electrode is placed into the solution and the other attached to the collector [5, 12]. A high voltage electrostatic field is used in this method that ejects a liquid jet through a spinneret. This field is charged the surface of a polymer solution, therefore droplets of the polymer are migrated to the collector [10]. As the intensity of the electric field increased enough to

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overcome the surface tension of the droplet, a charged jet of polymer solution is ejected [5, 12-16]. The electric force is affected the route of the charged jet [10]. The jet undergoes an elongation process [11] that cause to extend through spiraling loops. Therefore, diameter of the loops increase and the jet grows longer and thinner [13-16]. While the jet travels, the solvent is evaporated, a dried polymer fiber was attached on the collector [5, 12, 17, 18].

The electrospinning method is a simple and widely using method to fabricate nanofiber from a polymer solution. However, a few researches were purposed to reinforce of dental materials especially composite resins [5, 10, 12, 19, 20].

The aim of this study was to investigate effect of various mass fractions of electrospun Nylon 6 (N6) nanofibers to mechanical properties of Bis-GMA/TEGDMA DCR matrix.

2. MATERIALS AND METHODS

2.1. Materials

Nylon 6 (N6; catalog number 18, 111-0; Sigma-AldrichCo. LLC., Steinheim, Germany) and the solvent 1,1,1,3,3,3-hexa-fluoro-2-propanol (HFIP) were used in the study. Bisphenol A-Glycidyl methacrylate (BisGMA; Sigma-AldrichCo. LLC.) and tri-(ethylene glycol) dimethacrylate (TEGDMA; Sigma-AldrichCo. LLC.) monomers*,* camphorquinone (CQ; Sigma-AldrichCo. LLC.) used as photo-initiator and co-initiator ethyl-4 (N, N'-dimethylamino) benzoate (4EDMAB; Sigma-Aldrich Co. LLC.) were used for photo curing composite resin.

2.2. Electrospinning

A solution was prepared using 10% (mass fraction) N6 dissolved in HFIP. Positive high voltage was applied to solution using a copper wire in the electrospinning process. The solution was stirred at room temperature until acquired a homogeneous clear liquid. The solution was poured in 3ml syringes fitted with metallic needles of 0.8mm of inner diameter. Syringe with homogeneous solutions was inserted horizontally on the syringe pump (NE-1600, New Era Pump Systems Inc., NY, USA). The polymer solutions were pumped (feed rate: 1mL/h) during electrospinning process. High voltage power supply (ES30, Gamma High Voltage Research, Ormond Beach, FL, USA) was used to apply 15 kV to the metal needle tip. Distance between the needle tip and the collector was set at 10cm. N6 nanofibers were deposited on the collector that was covered with electrically grounded aluminum foil. (Figure **1**) On the way from tip to the cylindrical metal collector the solvents evaporated. Produced N6 nanofibers were observed by scanning electron microscope (SEM) (FEI Quanta 400F). Morphology of the N6 nanofibers (e.g. uniform diameter, smoothness and bead-free formation) were checked.

2.3. Fabrication of Specimens

A composite resin matrix was prepared by mixing 49.5% BisGMA, 49.5% TEGDMA, 0.2% CQ and 0.8% 4EDMAB which also constituted the control group. The nanofiber felt was cut into small pieces and soaked in the resin matrix. The soaked felt pieces were taken out of the resin matrix and photo cured for 2 minutes using curing light (QHL 75; Dentsply, Milford, DE, USA).

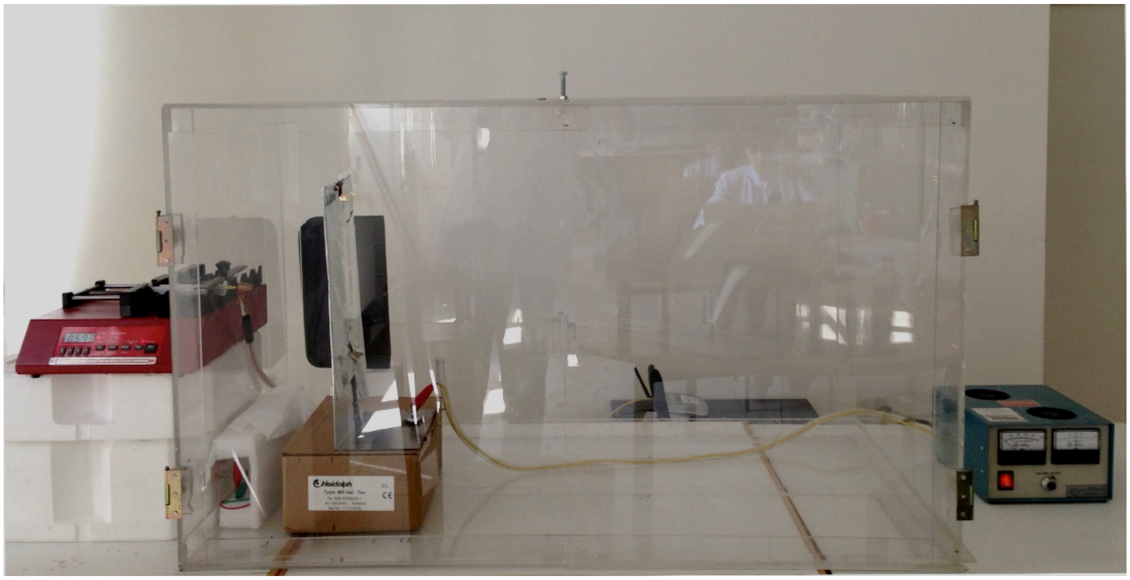

Figure 1: Electrospinning device.

Polymerized pieces were milled into powder with oscillating mill. The powders were added into the composite resin matrix at different mass fractions (1%, 2%, and 3%). The specimens (2mm x 2mm x 25mm) were prepared using a teflon mold. Neat resin was poured into the teflon mold for control group. Ultrasonic vibration was performed to remove trapped air bubbles in the immersed pieces. Then the specimens were photo cured for 2 minutes with curing light (QHL 75). Before the mechanical test, the specimens were immersed in distilled water at 37°C for 24 hours. All sides of the specimens were polished in a longitudinal direction using 2400 grit silicon carbide paper with water coolant. Eight specimens were prepared for each group and mechanical test was performed.

2.4. Mechanical Properties

Three point bending jig with a 20mm span was used to fracture the specimens on a computer-controlled Universal Testing Machine (LRX, Lloyd Instruments LTD., Fareham Hants**,** England). (Figure **2**) Cross-head speed was set 1mm/min to record stress strain curves. Eight specimens were tested in each group and the flexural strength (Fs), flexural modulus (E_Y) and work of fracture (WOF) of nanofiber reinforced BisGMA/ TEGDMA composite were investigated. Calculations were made using the following formulas: $FS = 3PL/2WT^2$; $E_Y = (P/d)(L^3/4WT^3)$; $WOF = A/(WT)$.

Figure 2: Three point bending test was perform using computer-controlled Universal Testing Machine at a cross-head speed of 1mm/min.

Table 1: Descriptive Statistical Analysis of Fs, E_Y and WOF Results

	Group	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
						Lower Bound	Upper Bound		
Fs	Control	8	66,32	18,04	6,38	52,24	81,41	50,67	104,30
	N6 1%	8	78,63	21,8	7,73	60,34	96,92	45,13	107,64
	N6 2%	8	82,67	20,1	7,12	65,84	99,51	63,70	116,41
	N6 3%	8	92,65	19,9	6,00	78,47	106,84	66,24	115,57
	Total	32	80,07	20,74	3,67	72,60	87,55	45,13	116,41
E_Y	Control	8	701,71	195,30	69,05	538,44	865,00	412,20	1021,60
	N6 1%	8	1728,78	518,248	183,23	1296,51	2162,05	1069,20	2703,02
	N6 2%	8	1811,18	1266,64	447,83	752,25	2870,13	530,76	3666,68
	N6 3%	8	1425,64	752,62	266,09	796,43	2054,86	654,05	2746,04
	Total	32	1416,83	869,88	153,77	1103,21	1730,46	412,20	3666,68
WOF	Control	8	10,84	2,48	5,24	70,26	93,97	52,57	108,62
	N6 1%	8	5,21	1,48	3,90	91,38	109,01	86,54	119,41
	N6 2%	8	7,39	4,23	5,51	99,74	124,66	90,03	135,66
	N6 3%	8	8,66	3,52	5,14	82,98	106,22	77,32	133,77
	Total	32	8,03	3,61	0,25	5,11	6,09	1,68	14,96

Where P is the load at fracture, L is the distance between two supports (which was set to be 20mm), W is the width of the specimen, T is the thickness of the specimen, and d is the deflection, in millimeters, at load P. In the formula of WOF, A is the area under the load-displacement curve, which is the work done by the applied load to deflect and fracture the specimen. With the unit of a being J (Joules), the unit of WOF (or fracture resistance) is J/m^2 or more conveniently, kJ/m^2.

The analysis of variance (ANOVA) was used for the statistical analysis of the acquired data. Tukey's multiple tests were used to compare the Fs, Ey and WOF means and significant levels were considered at p values ≤ 5. The level of confidence was established at α = 5%. Statistical analysis was performed using statistical package SPSS 11.5 for Windows (SPSS Inc, Chicago, USA).

3. RESULTS

The mechanical properties of different mass fractions of the N6 nanofiber reinforced dental composites (NRDC) were tested. A standard three-point bending test method was used and the results are shown in Table **1**. Fs, Ey and WOF values of the specimens were calculated and evaluated with statistical analysis.

Fs results of the control, and N6 1%, 2%, and 3% nanofiber filled composite groups (mean ± standard deviation, n = 8) were 66,32 MPa (±18.04), 78,63 MPa (±21,8), 82,67 MPa (±20,1), 92,65 MPa (±19,9) respectively. Fs results were shown increasing trend going to the highest mass fraction of N6 nanofiber, but these findings are not meant statistically significant. (ANOVA, p>0,05) (Figure **3**).

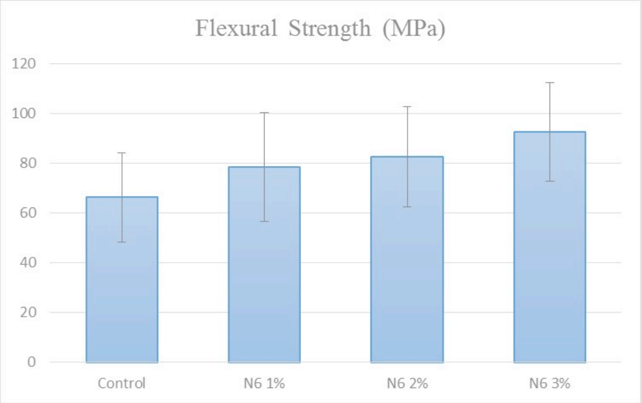

Figure 3: Comparison of Flexural Strength (Fs) results.

E_Y results of the control, and N6 1%, 2%, and 3% nanofiber filled composite groups (mean ± standard deviation, n = 8) were 701,71 (±195,30), 1728,78 MPa (±518,248), 1811,18 MPa (±1266,64), 1425,64 MPa (±752,62) respectively. E_Y results of the N6 2% nanofiber filled composite resin group was shown highest value among the groups and difference corresponding to control group was statistically significant. (Tukey, p=0,041< α=0,05) (Figure **4**).

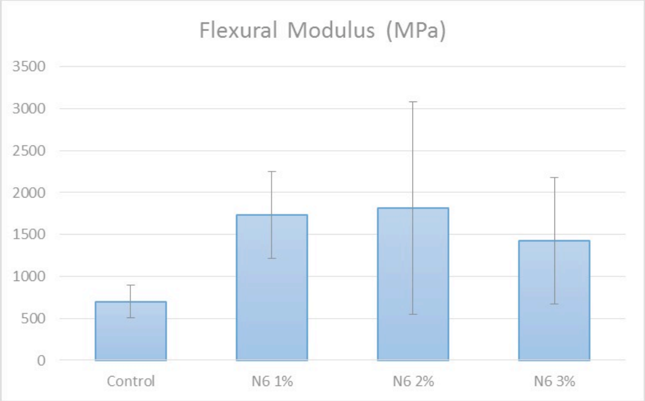

Figure 4: Comparison of Flexural Modulus (E_Y) results.

WOF results of the control, and N6 1%, 2%, and 3% nanofiber filled composite groups (mean ± standard deviation, n = 8) were 10,84 kJ/m^2 (±2.48), 5,21 kJ/m^2 (±1,48), 7,39 kJ/m^2 (±4,23), 8,66 kJ/m2 (±3,52) respectively. Difference of WOF results between the control and N6 1% nanofiber filled composite groups was statistically significant. (Tukey, p=0,006< α=0,05) (Figure **5**).

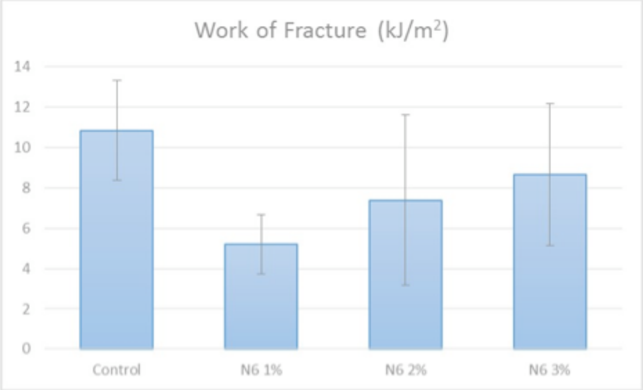

Figure 5: Comparison of Work of Fracture (WOF) results.

4. DISCUSSION

The DCRs are biocompatible and esthetic restorative material that are used frequently during dental therapy by clinicians [6, 12]. In spite of the advantages of the DCRs, mechanical properties of the resin are need to improve for long term successful results. Therefore, many fillers such as glass/ceramic fillers, glass fibers, whiskers, and nano particles are added into the resin matrix. Besides, a few amounts of the nanofibers could provide significant effect on mechanical properties of the resin. Because, the nanofibers have high surface area to volume ratio, small pore dimensions, good interfacial properties, and strong interactions between the resin matrix and other fillers, and bridging mechanism [8, 19, 21]. The nanofibers remain intact in the micro-crack lines and it can resist the loads and deflect the cracks. By this means, opening of the cracks can be prevented from the crack bridging nanofibers [22].

The electrospinning is widely used fabrication type of the nanofibers because of simple and suitable for polymers, ceramics and metals [21]. Many authors [3, 5, 6, 12, 20, 22-25] conducted to evaluate some properties of electrospun NRDCs. Hence, the clinicians' expectations grow up for using of clinically.

Key phenomenon of electrospinning process is bending instability. This may cause to elongate electrospinning jet up to 100,000 times less than 0.1 second [13]. High aspect draw ratio can generate extended chains. This formation of electrospun nanofibers could strengthen the resins [5].

Tian *et al*. [12] evaluated mechanical properties of N6 NRDCs. They had put 1%, 2%, 4% and 8% mass fractions of N6 nanofibers into the resin matrix. The authors reported that 1% and 2% N6 nanofibers successfully reinforced the DCR. On the contrary, 4% and 8% N6 NRDCs did not display same reinforcement significantly. They explained that use of high ratios of nanofibers can cause an increase in defect formation between the nanofiber and the matrix, and the nanofiber bonding to the matrix could be negatively affected.

Fong [5] investigated mechanical properties of N6 NRDC with various mass fractions. 5% N6 NRDC group showed significant increase in the mechanical properties compared to 2.5% N6 NRDC group. 7.5% N6 NRDC group did not show a significant increase relative to control group. The researcher explained that adhesion between the resin matrix and nanofibers might have been insufficient. SEM images of the fracture surfaces was proven that. Pulled-out nanofibers with any resin remnants were observed on the surfaces. Also, control group was showed large fracture steps, while in the N6 NRDC groups many fracture steps were seen.

The reinforcement of BisGMA/TEGDMA dental resins with various mass fractions of nano-fibrillar silicate was studied by Tian *et al*. [7]. Impregnation of small mass fractions of the nano-fibrillar silicate into the resin matrix improved the mechanical properties substantially. However, the authors concluded that larger mass fraction did not enhance mechanical properties, and may even have reduced the mechanical properties. Therefore, the small mass fractions (1%, 2% and 3%) of the N6 nanofibers were added into the resin matrix for this research.

Core-shell nanofibers could be used to improve mechanical properties of DCRs. Aim of the use of this type of nanofibers is to enhance adhesion between the shell and the resin matrix. In addition, it could be reinforced the DCRs *via* strong a core [6, 23, 24]. Lin *et al*. [6] reported the fabrication of PAN core-PMMA shell structured nanofibers by electrospinning and used PAN-PMMA, PAN and PMMA nanofibers at different mass fractions (2.5%, 5%, 7.5%, 10%). The researchers stated that the PAN-PMMA nanofibers reinforced the composite resin matrix before the mass fraction increased to 7.5wt%. However, the mechanical properties of the composite resin with 10% mass fraction of PAN-PMMA nanofibers were decreased significantly. PAN nanofibers improved the mechanical properties of the resin, but mass fraction of PAN nanofibers increased from 5% to 10% the mechanical properties decreased remarkably. On the contrary, mechanical properties of PMMA nanofiber reinforced composite resin group showed lower values than neat resin.

The researchers reported that the interfacial bonding between PAN-PMMA nanofiber and resin matrix was stronger than those of PAN and PMMA. The mechanical properties were improved due to better interfacial adhesion between resin matrix and PAN-PMMA nanofiber. The more surface area of the nanofibers, the more chance to result in defects, which is because further improvements in mechanical properties of the composites *via* increasing the amount of nanofibers might be limited.

Similarly Sun *et al*. [24] used PAN core-PMMA shell nanofibers to reinforce BisGMA/TEGDMA composite resins. The investigators reported an improvement in the mechanical properties of the composite. They used post-draw process to nanofibers and confirmed this treatment a useful method for significantly increasing the tensile strength and tensile modulus of nanofiber membranes. SEM analysis of the fracture surfaces showed that post-draw PAN-PMMA nanofibers were perfectly bonded to the resin matrix. Semi-interpenetrating network (Semi-IPN) formation enhanced adhesion between the resin matrix and the nanofibers. Thus, post-draw PAN-PMMA showed better mechanical properties compared to PAN-PMMA NRDCs.

Cheng *et al*. [23] studied sodium fluoride (NaF) loaded PAN(core)-PMMA(shell) nanofibers reinforced DCRs. 0.8% and 1% nanocrystalline NaF were added to the core of the PAN-PMMA nanofibers. The researchers concluded that this loading process did not damage the core-shell structure of the PAN-PMMA nanofibers. However, mechanical properties of the DCRs were enhanced *via* superior adhesion between the resin matrix and nanofibers. Also, NaF loaded PAN-PMMA nanofibers can have the added benefit of releasing fluoride that prevents caries. Electrospinning process of the NaF loaded PAN-PMMA nanofiber affects thickness of the shell which was increased, mechanical properties of the resin may be decreased.

In the current study, the specimens were prepared using BisGMA/TEGDMA soaked nanofiber powder and extra amount of BisGMA/TEGDMA monomers. The small diameter of nanofiber particles provided a high ratio of surface area to volume, that could improve inter-molecular hydrogen bonding between the filler of N6 nanofiber and the matrix of BisGMA/TEGDMA resin [5]. The three point bending test results of present study agreed that mechanical properties of the composite resins could be increased small amount of BisGMA/TEGDMA soaked N6 nanofiber powder.

WOF result of the control group was better than N6 NRDC groups but the difference between the control group and 1% NRDC group was statistically significant. E_Y result of the 3% N6 NRDC group was lower than other N6 NRDC groups. Increasing mass fraction of the nanofiber can lead to defect formation which obviously weakens the composite. But, addition of the 3% N6 nanofiber into the DCR was reinforced mechanical properties of the resin.

CONCLUSIONS

The small amount of nanofibers increase ratio of surface area to volume which could enhance the inter-molecular hydrogen bonding between the N6 nanofiber and the resin matrix. This study was indicated that Fs and E_Y results of the N6 NRDCs could all be increased after impregnating relatively small amount of the N6 nanofibers. Addition of the N6 nanofibers into the DCRs was decreased WOF results of the NRDCs, but differences between the control group and NRDC groups were not statistically significant.

The current study shows that a mechanical reinforcement is possible to add BisGMA/TEGDMA soaked nanofiber powder into the DCRs. This NRDCs can be used in dental practice for dental restorations. However, further analyses such as thermal property, dimension stability, biocompatibility and surface hardness need to prove the N6 NRDCs which are appropriate for clinical usage.

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