Evaluating the Effect of Addition of Titanium Dioxide Nanoparticle on Some Physical Properties of Flowable Composite Resin

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Abstract

Objective: The flowable dental composite resins were introduced to the dental specialty because of the advantages they possess over the conventional composite resins. The use of nanotechnology in the dentistry field is one of the growing innovations in recent years. The aim of the present study was to evaluate certain physical properties of flowable dental composite after incorporation of titanium dioxide nanoparticles (TiO2NPs).

Materials and Method: In the present study, TiO2NPs at 1.25 % and 2.5% concentrations were added to flowable composite, while the unmodified composite was used as control. Then the physical properties of the control and modified composite resins, including flowablity, radiopacity and water sorption and solubility were tested. Data were analyzed with One way ANOVA, using SPSS 20.

Results: The results showed that there was statistically significant difference among the tested groups regarding flowability and radiopacity ($P<0.05$). In addition, there was no significant difference among control group and TiO2 modified groups regarding water sorption and solubility.

Conclusion: Based on the results of the present study, a flowable dental composite was successfully reinforced with TiO2. Incorporation of small weight percentages of this nanofiller exhibited properties similar to the control material regarding water sorption and solubility. The flowability was slightly reduced and radiopacity of the reinforced composites was increased, these changes were acceptable for clinical applications and below ISO standards limits.

Keywords: *Flowable Composites, Titanium Dioxide nanoparticles, flowability, radiopacity, water sorption and solubility.*

Introduction

Currently composite resins are materials used in restorative dentistry, since there are many revolution and improvements such as use of different novel particles, flowable composite resin because of their advantages over the conventional composite resins were introduced to the dental specialty. These advantages includes, simple application technique, easy handling properties, increased flowability, better adaptation to the internal cavity wall and higher elasticity, in addition, the use of flowable composites resins reduces the amount of the cavity preparation and is recommended for minimal invasive dentistry (1).

One of the most important innovations in the dentistry field in recent years is the use of nanotechnology. The mechanical, physical and optical properties of conventional composite resins have been improved by addition of inorganic nanoparticles such as silver, zinc, titanium dioxide and silica (2).

Flowability is used to describe how quickly materials flow in a certain period of time, whereas viscosity is the material's resistance to flow. Dental composites are viscoelastic materials. They share criteria of both viscous materials (e.g., oils) and elastic materials (e.g., metals).

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Radiopacity of dental restorative materials is an important indicator for accurate diagnosis and treatment planning. It is essential to use materials with adequate radiopacity in order to distinguish them from the natural tooth structures. Moreover, dental restorations should be radiopaque enough to detect overhanging margins, recurrent caries, restoration contour, proximal contacts (3).

The sorption and solubility properties are critical properties regarding biocompatibility concerns of releasing monomer and in relation to the stability of the composites due to degradation from the uptake of solvents and the wash-out of ingredients of materials (4)

Most of the studies involving addition of nanoparticles to dental composites resins have mainly focused on their anti-bacterial effects and the information regarding their physico-mechanical properties are limited. TiO2 are fine, non-toxic, chemically stable, and exhibits a high photocatalytic effect in addition to their high biocompatibility and pleasant color. TiO2NPs have large surface area that facilitates load transfer from resin matrix to nanoparticles thereby resulting in better mechanical properties of the reinforced composites (5).

Mohammed et al (2019) studied the effect of TiO2NPs on physico-mechanical properties of flowable dental composite resins by adding TiO2NPs at 1%, 2% and 3% to Tetric N Flow composite, while the unmodified composite was used as control. The developed composite was studies for functional and structural properties using FTIR, which indicated no change in the functional and structural characteristics (6).

The aim of the present study was to evaluate the effect of TiO2NPs, on some physical properties of flowable composite resins.

Method

In this study, the flowability, radiopacity and water sorption and solubility of a conventional flowable composite resin and composite resins reinforced with TiO2NPs were evaluated. For each test a total of 18 samples were evaluated. Commercially available flowable microhybrid composite resin (as control group) was used shade (A2). This material is based on dimethacrylate paste (Bis-GMA and Triethylene glycoldimethacrylate TEGDMA), without inorganic fillers. mixed with TiO2NPs 98% Purity and particle size of 50 nm at concentrations of 1.25% and 2.5 %.

Mixing was done by using a dental Micro motor, with a lentulo spiral-paste carrier #4 attached. The lentulo spiral was immersed in composite resin material – TiO2NPs, poured into a 2 ml pre-darkened syringe tube.

Such mixing was performed directly before preparation of samples for each test. Electronic balance was used to weight the percentage of nanoparticles, after one hour mixing; the mixture was injected into metal molds Curing was done by exposure to LED at 1,650 mW/cm2 for 40 seconds with a light guide held perpendicularly and within 2 mm of the material surface. Control samples were prepared and compared with two sets of samples with TiO2NPs at 1.25% and 2.5%.

The flowability testing method used in this study was according to ADA guidelines for evaluation of endodontic sealing materials (7). To evaluate the flowability of the reinforced composites, a simple test using the Gillmore needle apparatus was used. The quantity of 0.1 mL of control and each reinforced composite was dispensed between two thin glass coverslips 50 x 50 mm and 1mm thick. Flowability was evaluated by comparing the composite disc diameters after they have been sandwiched between two glass coverslips, subjected to constant weight (454 g) for 30 s and light cured for 40 s.

For radiopacity test 6 discs 15±1mm in diameter and 1± 0.1mm thick were prepared for each group. The specimens and the aluminum stepwedge were placed in the center of the film and irradiated with X-rays at (65±5) kV at a target film distance of 400 mm for 0.4 seconds at 10 mA. After developing and fixing the film, the density of the image of the specimen was compared with that of the aluminum standard using the densitometer. For Water sorption and solubility test 6 discs 15±1mm in diameter and 0.5± 0.1mm thick samples were prepared for each group using a metal molds and tested according to ADA specification (8)

Statistical Analysis

The statistical analysis was performed on SPSS program version 20.0, the descriptive statistic was done for tested groups, for verification the difference among groups analysis of variance (One-way ANOVA) with Tukey post-test.

Results

Flowability test

Table 1 present the means and standard deviations of flowability values in conventional composite resin and composite resins reinforced with TiO2NPs at different percentages.

Based on table 1, the Flowability of control group and 1.25% and 2.5% groups were 2.6933 cm, 2.6083 cm and 2.5067 cm respectively, The Results of Table 1 revealed significant differences among the tested groups. But the difference is acceptable within the ISO standard.

Table 1: Mean values (mm), standard deviations, standard errors and 95% confidence intervals for Flowability data.

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
(C)	6	2.6933a	.00816	.00333	2.6848	2.7019	2.68	2.70
1.25%	6	2.6083b	.00983	.00401	2.5980	2.6187	2.60	2.62
2.5%	6	2.5067c	.01211	.00494	2.4940	2.5194	2.50	2.53
Total	18	2.6028	.07910	.01864	2.5634	2.6421	2.50	2.70

Note: Means with different letter indicates statistically significant difference (P<0.05).

Radiopacity test

Radiopacity mean values of the tested materials are presented in Table (2). As recommended by ISO $^{(9)}$. 2.5% group showed the highest radiopacity values 2.4760 E.q. Al thickness/mm mean value followed by 1.25% group of 2.3900 E.q. Al thickness/mm mean value of radiopacity, and 2.2833 E.q. Al thickness/mm mean value for Control group with statistical significant difference among the tested groups.

Table 2: Mean values (mm), standard deviations, standard errors and 95% confidence intervals for Radiopacity.

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
control	6	2.2833a	.00816	.00333	2.2748	2.2919	2.27	2.29
1.25%	6	2.3900b	.00894	.00365	2.3806	2.3994	2.38	2.40
2.5%	6	2.4760c	.01131	.00462	2.4641	2.4879	2.46	2.49
Total	18	2.3831	.08159	.01923	2.3425	2.4237	2.27	2.49

Note: Means with different letter indicates statistically significant difference (P<0.05).

Water Sorption test

Water sorption mean values are presented in Table (3) revealed no significant differences among the tested groups. Control group showed the highest Water sorption values 1.456283 µg/mm^3 mean value, followed by 1.25% group with -1.029533 µg/mm^3 mean value Water sorption, and .802833 µg/mm^3 mean value of 2.5% group but the difference was not significant statistically.

Table 3: Mean values (mm), standard deviations, standard errors and 95% confidence intervals for Water sorption data test.

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
control	6	-1.456283a	1.2018703	.4906615	-2.717569	-.194998	-2.8037	.8131
1.25%	6	-1.029533a	1.3579421	.5543775	-2.454606	.395539	-1.8691	1.6260
2.5%	6	-.802833a	1.6504075	.6737760	-2.534830	.929163	-1.6260	2.5000
Total	18	-1.096217	1.3586916	.3202467	-1.771878	-.420555	-2.8037	2.5000

Note: Means with same letter indicates statistically no significant difference.

Water Solubility

Solubility mean values are presented in Table (4). Test results revealed no significant differences among the tested groups. Control group showed the highest solubility values 2.516583 µg/mm^3 mean value, followed by 1.25% group with 2.048467µg/mm^3 mean value solubility, and 1.386167µg/mm^3 mean value of 2.5% group with no statistical significant difference among the tested groups.

Table 4: Mean values (mm), standard deviations, standard errors and 95% confidence intervals for Water solubility data test.

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
control	6	2.516583a	.9013909	.3679913	1.570632	3.462535	.9345	3.2601
1.25%	6	2.048467a	1.1576720	.4726176	.833564	3.263369	.8130	3.8530
2.5%	6	1.386167a	1.1753861	.4798494	.152675	2.619659	.0000	3.4390
Total	18	1.983739	1.1257010	.2653303	1.423941	2.543537	.0000	3.8530

Note: Means with same letter indicates statistically no significant difference.

Discussion

The flowability of floweble composite resin decreased slightly with addition of TiO2NPs at 1.25% and 2.5% and the results are within the ISO standards, these results are in agreement with previous study reported that it is possible to increase the nanofiller content due to their small particle sizes. This will result in minimum reduction in flowability as well as improvement in the physical properties of the nano-composites (10).

In the present study the most commonly used monomer) BisGMA and TEGDMA were used, The advantages of using BisGMA over other monomers are less shrinkage, higher modulus and reduce toxicity due to its lower volatility and diffusivity into tissue. Although, BisGMA possesses high strength and hardness, the drawback of this monomer is its high viscosity (low flow), because of hydrogen bonding interaction that occur between hydroxyl groups, which limits the incorporation of inorganic fillers and hence a low degree of conversion,Thus BisGMA diluted with other low-viscosity (high flow) monomer such as trimethylene-glycol-dimethacrylate (TEGDMA) (11) to enhance flowability of the used composite that allow incorporation of TiO2NPs that resulted in a little reduction of flowability of the tested flowable composite resin and the changes are within accepted range of ISO standards

The radiopacity of a restorative material is an important parameter for accurate diagnosis and treatment planning. Success or failure of the restorative material is highly dependent on radiographs. Radiopacity of composite restorations has an important role in detecting recurrent caries and distinguishing the restorations from the tooth structures (12).

The 2.5% composite showed a higher value of radiopacity. Followed by 1.25% group Al, and control group with statistical significant difference among the tested groups and met the ISO standard for dental materials radiopacity. All of them had radiopacity greater than enamel (1.77 – 2 mm Al). These results agree with previous studies that recommended that the composite radiopacity should be equal to or greater than that of the enamel (13).

The atomic number of the elements is the most important factor affecting the radiopacity of dental materials (14). Radiopacity of dental composites can be increased by incorporating a higher percentage of fillers with high atomic numbers (15), TiO2NPs atomic number is 22 and density of 4.506 $g/^{cm3}$ which is considered as a high atomic number and this explains the results of this study in which radiopacity of tested material increased with increasing percentage of TiO2NPs from 0% control group to 1.25% and 2.5 %..

The sorption and solubility properties are important regarding biocompatibility concerns of releasing monomer and in relation to the stability of the composites by avoiding degradation from the uptake of solvents and the wash-out of ingredients of materials (16). According to ISO standard (17), the maximum acceptable values of sorption and solubility for polymer-based restorative materials are 40 µg/mm3 and 7.5 µg/mm3 respectively. Sorption and solubility values for all samples were below the ISO standards limits so all investigated materials met the requirements of the ISO standard. The decreasing sorption and solubility with TiO2 content was not statistically significant. These results are in agreement with Robert et al (18) study who studied the effect of nanofillers on water sorption and solubility and concluded that the addition of nanofillers at low concentrations not change water sorption and solubility significantly since at lower concentrations there is no agglomeration of 50 nm TiO2NPs.

The improvement of both water sorption and solubility after addition of TiO2NPs might be attributed to numerous explanations such as nanofillers are insoluble in water so that the addition of TiO2NPs to the microhybrid flowable composite resin declines the solubility of composite resin (19).

Furthermore, titanium coupling agent incorporated in salinized TiO2NPs expands the adhesion between both resin matrix and filler particles which enhances composite resin properties and declines its water sorption and solubility (20).

Moreover, the reaction between resin (polar nature) and nanofillers certainly induce replacing the hydrophilic resin and minimizing the water uptake by decreasing this polarity through utilizing most active sites in the molecules of momomers, so the diffusivity of water particles through this material is greatly declined (21).

Conclusion

Based on the results, it appears incorporation of low concentration of nanofillers into conventional composite resins did not result in any changes in their water sorption and solubility; however, flowability and radiopacity of flowable were changed but the results are acceptable withen ISO standards .Therefore, it is suggested to add small amount of nano-fillers to composite resins to prevent problems such as discoloration of composite resin or other possible changes in other physical and chemical properties.

Ethical Clearance: The Research Ethical Committee at scientific research by ethical approval of both MOH and MOHSER in Iraq

Conflict of Interest: Non

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