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Effects of adding graphene oxide to composite resin: An in vitro study

Beatriz Márquez Villegas[1], Carolina Sámano Valencia[1], Jessica Campos-Delgado[2], América Rivera-Urbalejo[1,3], Erik Reyes Cervantes[4], Kelly Castro[5], Julia Flores-Tochihuitl[6]

1. *Benemérita Universidad Autónoma de Puebla, Facultad de Estomatología, Puebla, México.*
2. *Universidad de las Américas Puebla, Departamento de Ciencias Químico Biológicas, Puebla, México.*
3. *Benemérita Universidad Autónoma de Puebla, Grupo Ecología y sobrevivencia de microorganismos. LEMM, CICM, IC, BUAP, Puebla, México.*
4. *Benemérita Universidad Autónoma de Puebla, Dirección de innovación y transferencia de conocimiento, Puebla, México.*
5. *Instituto Nacional de Metrologia, Qualidade e Tecnologia. Rio de Janeiro, Brasil.*
6. *Benemérita Universidad Autónoma de Puebla, Facultad de Estomatología, Laboratorio Multidisciplinario, Puebla, México.*

ABSTRACT

Composite resin has been extensively used as a direct restorative material to restore posterior teeth due to its low cost and more complete preservation of the tooth than conventional resin. Nanohybrid resins are a popular alternative due to their apparently better mechanical strength and aesthetic properties compared to conventional resins. However, their clinical performance is still under evaluation. Adding small amounts of graphene oxide (GO) could improve the physicochemical properties of these resins. ***Aim:*** *The purpose of the current study was to evaluate the effects on the morphological and physicochemical properties of nanohybrid composite resins when GO is added to them.* ***Materials and Method:*** *Three kinds of composite resin (Universal Nanohybrid Restorer 3M Filtek Z250 XT) were evaluated: G0, without GO; G1, composite resin with 0.01 % GO; and G2, composite resin with 0.02 % GO. Surface morphology and physicochemical characterization were analyzed for all three groups. Data were submitted to analysis of variance, followed by post hoc Tukey's tests at a significance level of $p < 0.05$.* ***Results:*** *The addition of GO did not modify the surface of the composite resin compared to the control. The peak of the GO spectrum obtained by FTIR was very small and almost imperceptible due to the close combination of graphene molecules with the rest of the material. Flexural strength increased significantly in group G1, but decreased in G2 compared to the control group. Both GO concentrations resulted in a significant increase in resistance to wear compared to the control group.* ***Conclusions:*** *The addition of graphene oxide did not modify the surface morphology of the nanohybrid compound resin evaluated. However, it did produce changes in mechanical properties: resistance to wear increased significantly at both concentrations studied, while flexural strength changed in a concentration-dependent manner. These results suggest that low concentrations of GO may contribute to improving certain physicochemical properties of nanohybrid compound resins.*

Keywords: *composite resin - graphene oxide - dental restoration wear - flexural strength*

Efecto de la adición de óxido de grafeno a resina compuesta: estudio *in vitro*

RESUMEN

La resina compuesta se ha utilizado extensamente como material de restauración directa en el sector posterior debido a su bajo costo y una mayor preservación de la estructura dentaria. Las resinas nanohíbridas representan una alternativa atractiva por sus aparentemente mejores propiedades estéticas y resistencia con respecto a las resinas convencionales, sin embargo, su desempeño clínico continua en evaluación. En este sentido, la incorporación de cantidades pequeñas de óxido de grafeno (GO) podrían mejorar las propiedades fisicoquímicas de estas resinas. ***Objetivo:*** *El propósito de esta investigación fue evaluar los efectos de la incorporación del GO sobre las propiedades morfológicas superficiales y fisicoquímicas de una resina compuesta nanohíbrida.* ***Materiales and Métodos:*** *La resina compuesta (Restaurador Nanohíbrido Universal 3M Filtek Z250 XT) se evaluó en tres grupos: G0, sin GO; G1, resina compuesta con GO al 0,01 %; G2, resina compuesta con GO al 0,02 %. Se realizó la caracterización morfológica y fisicoquímica de la superficie en todos los grupos. Los datos se analizaron mediante análisis de varianza, seguido de pruebas post hoc de Tukey con un nivel de significancia de $p < 0.05$.* ***Resultados:*** *La incorporación de GO no modificó la superficie de la resina compuesta en comparación con el grupo control. El pico del espectro de GO obtenido por FTIR fue muy pequeño y casi imperceptible debido a la estrecha combinación de las moléculas de grafeno con el resto del material. En el grupo G1 se observó un aumento significativo de la resistencia a la flexión, mientras que en el grupo G2 se observó una disminución en comparación con el grupo control. La incorporación de ambas concentraciones de GO resultó en un aumento significativo de la resistencia al desgaste en comparación con el grupo control.* ***Conclusiones:*** *La incorporación de óxido de grafeno no modificó la morfología superficial de la resina compuesta nanohíbrida evaluada. Sin embargo, produjo cambios en sus propiedades mecánicas, aumentando significativamente la resistencia al desgaste en ambas concentraciones estudiadas y modificando la resistencia a la flexión de manera dependiente de la concentración. Estos resultados sugieren que bajas concentraciones de GO pueden mejorar determinadas propiedades fisicoquímicas de las resinas compuestas nanohíbridas.*

Palabras claves: *resina compuesta - óxido de grafeno - restauración dental - resistencia a la flexión.*

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Corresponding Author:
Julia Flores-Tochihuitl
julia.flores@correo.buap.mx

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INTRODUCTION

From a materials science perspective, many dental materials are classifiable as "composites", including glass ionomer (polyalkenoate) cements and some impression materials. However, majority usage within dentistry conventionally confines the term "composite" to materials that have a polymeric (organic) matrix and a particulate inorganic glass phase, possibly with the addition of fibers, which are known as resin composites or polymeric composites[1], or by other more specific terms such as dimethacrylate composites[2]. Composite resin as a direct restorative material has been extensively used to restore posterior teeth due to its low cost and more complete preservation of the tooth than conventional resins, as well as good clinical performance[1]. Nanohybrid composite resins are used for posterior restorations. In a complex class II cavity with cervical walls in dentin, the marginal adaptation is more challenging in relation to the cervical wall in enamel due to the bond to dentin being more complex[3]. In addition, composite shrinkage stresses associated with the masticatory loads can decrease the bond strength between the tooth and the composite resin[4].

Nanohybrid resin composites contain conventional particles mixed with nanometric fillers, so their performance is similar to that of nanofilled or micro-hybrid resins. The addition of well dispersed inorganic particles to a resin matrix is extremely effective for improving the performance of polymeric composite material, providing high initial polishing, better polish and gloss retention, and resistance to wear[5].

Restorative materials have evolved to conform to increasingly greater demands and higher standards, often strictly related to aesthetic aspects. Thus, composite resins have become the material of choice for the restoration of posterior teeth[6].

The most significant change in commercial composite resins in recent years has been modification in mineral filler content. The size of the fillers added to the matrix of new composite resins has been reduced, leading to nano-filled composite resins with improved properties. Several studies have reported that nanocomposite resins (nanohybrids or nano-filled) have better surface polish and lower abrasion resistance, and found that nano-filled composite resins wear out by detachment of particles or individual parts of nanoclusters, rather than by detachment of larger particles, resulting in a relatively smooth wear surface[5]. The Academy of Dental Materials designates discoloration, modulus of elasticity, fracture resistance, fatigue, occlusion resistance, and abrasion and friction wear measurements as the most important characteristics for material performance evaluation[6,7].

Graphene oxide (GO), an oxidized form of graphene, is a carbon nanomaterial formed of single-atom layers. Graphene is a two-dimensional layer of sp^2 hybridized carbon atoms organized in a hexagonal framework. Its unique properties and prominent features include high electrical conductivity, optimal mechanical properties, large surface area, low thermal expansion coefficient, and very high aspect ratio[8].

Graphene oxide consists of graphene sheets to which oxygen-containing functional groups are attached. It is produced by the chemical oxidation of cheaply available graphite. As it readily disperses in water and other polar solvents, it is a good alternative for applications that require hydrophilicity, unlike graphene[9-10]. Generally, GO is believed to have a graphene backbone where hydroxyl and epoxide groups are on the basal plane, and predominantly carboxyl and hydroxyl groups are on the edges. There is also inter-platelet hydrogen bonding through the alcohol and epoxide functional groups, which helps to maintain the stacked structure of GO[10-12].

The aim of this study is to observe the effects on the morphological, physicochemical and mechanical properties of composite resin when GO is added to it in two different concentrations.

MATERIALS AND METHOD

This *in vitro* study with the was registered with the research committee of the School of Stomatology, Benemérita Universidad Autónoma de Puebla (registration code 202320). It is important to mention that prior to this analysis, a pilot test was carried out in which concentrations of 2%, 1% and 0.1% GO were added to the resin and tested. These concentrations were discarded because they caused a drastic change in color and a decrease in hardness in the flexural test.

Specimen preparation

In this *in vitro* study, GO (synthesized from graphite using the modified Hummers method[13], provided by

INMETRO, Brazil) was mixed with composite resin (Universal Nanohybrid Restorer 3M TM Filtek TMZ250 XT) at concentrations of 0.01 wt % and 0.02 wt %. To prepare the chemical mixture, the GO was weighed and mixed manually for 15 min under red light with 1g composite resin until the mixture was homogenous (process modified from Alghyamah et al.[9]). The following study groups were prepared: control group (without GO), G1 containing 0.01 wt % GO, and G2 containing 0.02 wt % GO. It is worth noting that the addition of these concentrations did not modify the macroscopic appearance of the resin. The following specimens were prepared with bulk fill composite (Universal Nanohybrid Restorer 3M TM Filtek TMZ250 XT): 15 disks (5 mm in diameter and 2 mm in height) to measure wear resistance, and 15 rods (22 mm x 2 mm x 2 mm) to measure flexural strength.

The specimens were light-cured, first at the center and then at the ends, using an IVOCLAR halogen light lamp coupled to a Blue phase N MC system (100-240 V). The specimens were polished with 500 silicon carbide discs to remove the oxygen-inhibited resin layer resulting from storage in distilled water at 37 °C for 48 hours. A digital vernier caliper was used to re-evaluate their sizes.

Morphological characterization

The morphological characterization of the modified composites containing GO was done via Scanning Electron Microscopy (SEM) using a JEOL model JSM-6610LV[14]. Specimens were attached to a pin with double-sided carbon tape, and gold-sputtered (DENTON VACUUM, DESK V). SEM voltage ranged from 15 kV to 30 kV, and magnification ranged from 500x to 1000x.

Fourier Transform InfraRed Spectroscopy (FTIR)

FTIR is used to define the vibrational deformations of chemical bonds. GO-incorporated composite resins were characterized by attenuated total reflectance-Fourier transform infrared spectroscopy (BRUKER, model VERTEX 70) in the range of 4000 a 400 cm^{-1} according to a previously reported method[14]. Representative images and results are shown for independent experiments performed in triplicate.

Flexural test

Flexural strength was measured on 5 rod-shaped specimens (22 mm x 2 mm x 2 mm) per group. After storage in distilled water at 37°C for 48 hours, the specimens were polished using 500-grit silicon carbide discs to remove the oxygen-inhibited layer. The flexural test was performed with an Instron universal destructive tensile testing machine (Model 4411, Instron Corp., Cantan, MA, USA), for 3-point flexural tests at a span length of 14 mm. The flexural strength and elastic modulus were measured at a 1.0 mm/min crosshead speed (n = 5). During the test, the rods were supported at two points. The load was applied to the surface in front of the support points at a point equally distant from the first two points. Compression events were observed on the rod before it fractured[15,16]. One-way analysis of variance was used to determine the statistical differences of flexural strength among groups with Graph pad version 7 package.

Wear resistance test

Resistance to wear was measured on 5 disk-shaped specimens (5 mm in diameter × 2 mm thickness) per group. The specimens were light-cured with a bluephase C8 (Ivoclar Vivadent) light-curing unit for 20 s, and stored in distilled water at room temperature for 24 h for maximum water sorption[17,18].

Before abrading, the samples were thoroughly cleaned and dried. The initial weight of each sample was recorded on a precision electronic analytical balance (Ohaus, accurate to 0.0001 g). After being sanded, specimens were abraded using a Strong 210/108 micromotor and contra-angle at 2500 rpm for 30 seconds. A constant load of 500 mg was applied to the disc using CPT microbrushes (used in the dental prophylaxis procedure and manufactured with steel bristle heads and nylon plastic). At the end of each abrasion cycle, the samples were thoroughly cleaned, dried, and weighed again to determine the amount of abrasion (adapted from Ziaei et al.[8]).

Statistical analysis

The statistics consultant determined the sample size based on similar articles. The data are expressed as the mean ± SD of five independent experiments, unless otherwise noted. Statistical significance was determined by one-way analysis of variance with a post hoc test (Tukey) using Graph Pad (version 7.0). A p-value < 0.05 was considered statistically significant.

RESULTS

The purpose of this study was to observe the effects of adding minute amounts of GO to composite resins, specifically on surface morphology and mechanical characteristics such as flexural strength and resistance to wear, to study the properties of GO described by other authors. All the tests were performed *in vitro*, which does not simulate all the conditions of the oral cavity. Due to the conditions in our laboratory, it was difficult to compare the results obtained with previous studies.

Surface morphology analysis with scanning electron microscopy (SEM)

The surface morphology and the distribution of GO in the composite specimens were investigated by SEM. Homogeneous surfaces were observed during morphological analysis. Before the abrasion process, examination of specimen roughness clearly showed that they all shared the same properties, and no agglomerations were observed (Fig. 1).

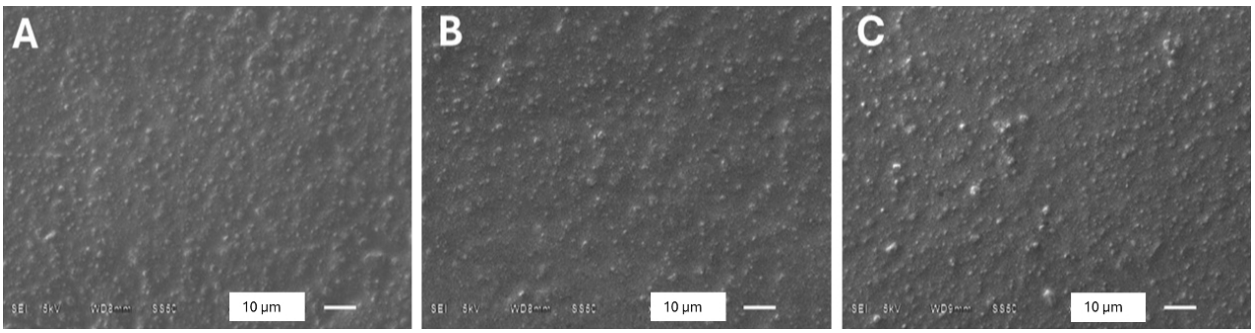

Fig. 1: Morphological characteristics of composite modified with GO. Micrographs show that the GO is uniformly distributed over the entire surface of the composite specimen in A) control, B) G1, and C) G2. Magnification 1000x.

Structural analysis by FT-IR

The FTIR spectra of the composite modified with GO were evaluated. The goal of FTIR analyses was to assess and compare the chemical structure of two series of resin-based samples. Peaks at 1000 and 1226 cm^{-1} that can be attributed to C-O vibrations, and peaks at 1026, 1150, 1630 and 1750 cm^{-1} reflecting C-OH, C-C and C=O groups can be observed in all samples, indicating that the addition of GO to G1 and G2 had no impact on the overall chemistry of the resins (Fig. 2).

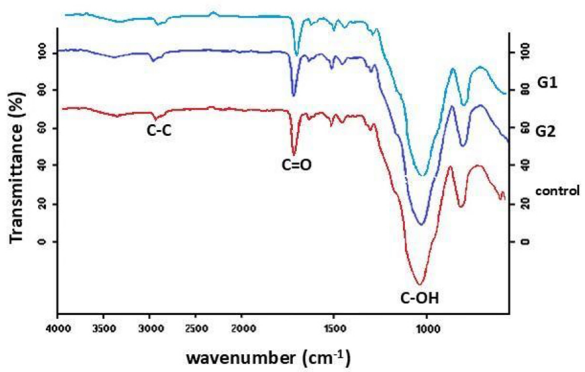

Fig. 2: FTIR spectra of the different groups: control, G1= 0.01% GO and G2= 0.02% GO.

Flexural test

One-way ANOVA showed $p<0.05$ for comparison of both G1 and G2 against the control group. Interestingly, there was a greater statistically significant difference for group G1 compared to group G2 ($p<0.001$) (Fig. 3A).

Wear resistance test

The lowest mass loss was observed in both group G1 (one-way ANOVA, $p<0.0001$) and G2 (one-way ANOVA, $p<0.0001$) compared to the control. It is important to highlight that G2 lost less mass than G1 (one-way ANOVA, $p<0.0001$) (Fig. 3B).

DISCUSSION

This study compared Filtek Z250 3M composite resins modified with 2 concentrations of GO. The aim was to investigate possible changes in morphology, chemical composition, and properties such as bending and wear.

SEM showed that there was no modification of surface morphology in specimens containing GO compared to the control (unmodified resin), therefore the addition of GO does not compromise the texture of the composite resin. This agrees with Guazzo et al.[19], who reported that GO does not modify the

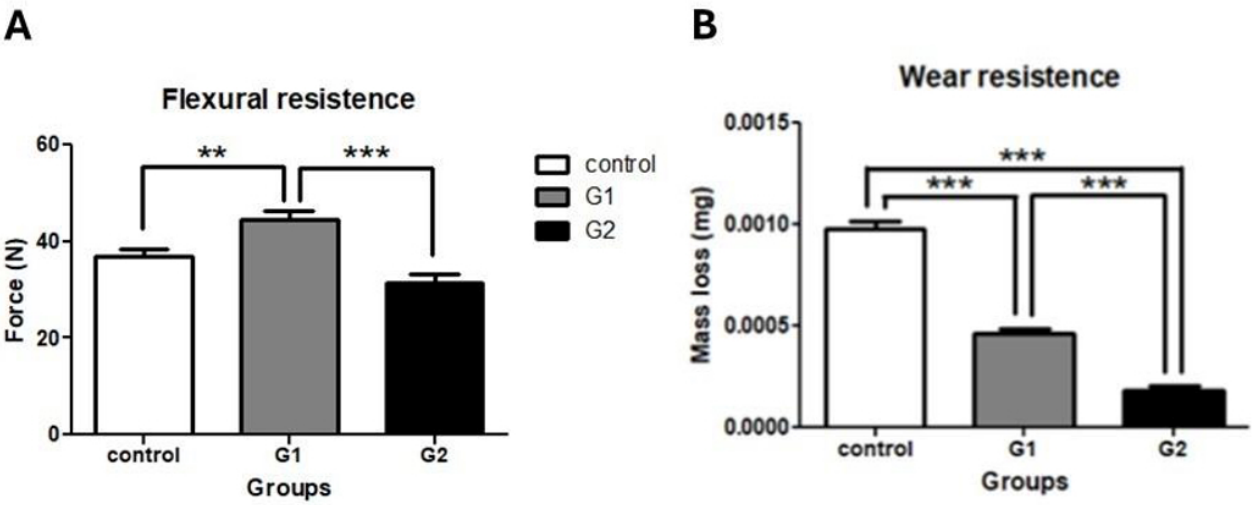

*Fig. 3: A) Flexural strength test showing statistically significant differences between groups G1 and G2. B) Wear resistance test compared to the control (n=5, one-way ANOVA, ** p<0.05 *** p<0.0001).*

texture of other materials. Thus, graphene-based materials may provide new, improved options or substitute materials currently used in dentistry.

FTIR analysis did not show different functional groups in the composite and modified resins, so it may be assumed that the addition of such small concentrations of GO did not alter the chemistry of the resin. Various papers report that the functional groups C-OH, C-C and C=O are present both in the materials containing graphene and in the composite resins (e.g., Agarwallaa et al.)[20]. Our result also agrees with Lee et al.[21], who published spectra of the GO with minimal signals corresponding to its functional groups, which are very small and almost imperceptible due to the close combination of graphene molecules with the rest of the material.

Flexural strength tests provide information about the ability of a material to flex before breaking. Group G1 had significantly greater compressive strength than the control group ($p< 0.05$). Increased resistance in the presence of GO agrees with Lee et al.[21], who added nano-graphene oxide (nGO) to PMMA-based acrylic resin, and found that it increased resistance by 0.5% compared to the control group. However, group G2 had significantly lower resistance ($p<0.001$), suggesting that the presence of a greater amount of GO could weaken the composite resin and decrease its resistance. It would thus be advisable to perform further tests using different percentages of GO, different techniques for adding GO to the resin, and different solvents to integrate it into the resin.

The wear test measured mass loss from specimens subjected to brushing. The presence of GO in the composite resin increased its resistance to wear with brushing, suggesting that when used as a restoration material, it would undergo less wear from daily toothbrushing. This agrees with Turssi et al[22], who reported that larger filler particles, the distance between the filler particles, and the amount of filling by weight are related to greater wear resistance of the resin. In addition, the resistance capacity that GO provides to other materials such as acrylic resins, adhesives, implant coatings, scaffolds, etc. has already been very well reported. Similarly, Dubey et al.[23] and Bitounis et al.[24] tested wear in various materials for tissue engineering and modified acrylic resins, concluding that GO provides the materials with greater resistance to bending and wear. The wear mechanism of a dental material consists of a series of intricate steps that can lead to loss of shape, increased roughness, pigmentation, and plaque buildup[25]. Therefore, to understand its limitations and indications, it is necessary to examine the restorative materials. In this approach, *in vitro* brush abrasion testing can be used to compare the abrasion resistance of various types of materials and determine which type of material would be best for use as a restorative treatment.

CONCLUSION

The addition of GO to Filtek Z250 XT composite resin resulted in a homogeneous distribution of the nanomaterial, without evidence of particle

agglomeration, as observed by SEM analysis. Furthermore, FTIR analysis indicated that the addition of GO did not significantly alter the overall chemical structure of the resin matrix.

The addition of low concentrations of GO influenced the mechanical performance of the composite resin. Both GO concentrations significantly improved wear resistance compared to the control group. Regarding flexural strength, the addition of 0.01 wt % GO resulted in a significant improvement, whereas 0.02 wt % GO led to a reduction in this property. These findings suggest that the effect of GO on the mechanical behavior of the composite is concentration-dependent.

Among the tested formulations, the composite containing 0.01 wt % GO exhibited the most favorable balance between flexural strength and wear resistance, indicating that low concentrations of GO may provide a promising strategy for enhancing selected properties of nanohybrid composite resins.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article.

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