

# Radiopacity of modified microhybrid composite resin: (An in vitro study)

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## ABSTRACT

**Background:** The aim of this study was to measure the radiopacity (RO) of modified microhybrid composite resins by adding 2 types of nanofillers (Zinc Oxide and Calcium Carbonate) in two concentrations 3% and 5% and comparing them to unmodified microhybrid composite resins and to nanofilled composite resin.

**Materials and Methods:** Two types of composite resin were used (Microhybrid composite MH Quadrent anterior shine and Nanofilled composite resin Filtek Z350 XT), for each tested group five disk-shaped specimens (1-mm-thick and 15 mm diameter) were fabricated. The material samples were radiographed together with the aluminum step wedge. The density of the specimens was determined with a transmission densitometer and was expressed in term of equivalent thickness of aluminum. Data analyzed by one-way ANOVA.

**Results:** The radiopacity (RO) values of the tested group ranged between (0.9293- 2.6242 Eq. Al thickness) and there were significant differences among them. Nanofilled composite resin Filtek Z350 XT showed the highest value of RO while unmodified Microhybrid composite MH Quadrent anterior shine showed the lowest value of RO.

**Conclusion:** The addition of 3% of both the ZnO and CaCO<sub>3</sub> nanofillers fillers to microhybrid composite significantly increased the RO, while the addition of 5% of CaCO<sub>3</sub> and ZnO nanofillers to microhybrid composite showed non-significant increase in the RO of the composite.

**Key words:** Resin composite, Radiopacity, Aluminum, Densitometer. (J Bagh Coll Dentistry 2013; 25(Special Issue 1):18-22).

## INTRODUCTION

The current trend in modern resin based composites (RBCs) of minimizing filler size whilst aiming to improve the filler loading has sought to optimize the resultant mechano-physical properties and clinical performance. The introduction of so-called 'nanofilled' and 'nano-hybrid' materials therefore appears a logical continuation of this trend. By definition, a 'nano-material' possesses components and/or structural features, such as fibres or particles, with at least one dimension in the range of 1-100nm and subsequently demonstrates novel and distinct properties <sup>(1,2)</sup>

One of the most desirable properties of any dental restorative materials is radiopacity, a property that facilitates the radiographic diagnoses adjacent to dental composites and enables better radiographic detection of secondary caries which is the cause for up to half of all operative dentistry procedures performed on adults. Furthermore, radiopaque materials enable the clinician to evaluate restoration integrity at following recall appointments, to detect voids, secondary caries, overhangs and open margins <sup>(3-5)</sup>.

Quadrent anterior shine is a microhybrid composite resin commonly used for anterior teeth. It had a low radiopacity. To enhance its radiopacity and to be use it for posterior teeth, certain modification should be investigated. Accordingly this study was designed to evaluate the radiopacity of previously mentioned composite after the addition of nano-sized fillers of Calcium carbonate and Zinc Oxide in two different concentrations.

Further studies to evaluate the other properties (other physical properties, mechanical and antibacterial properties) are in progress, and will be published as soon they are completed.

## MATERIALS AND METHODS

### Materials

Two commercial composite resins (Microhybrid composite MH Quadrent anterior shine, Nanofilled composite resin Filtek Z350 XT) were used in this study. Two types of coated nanofillers (Calcium carbonate and Zinc oxide) both were added to the microhybrid composite. The commercial name, composition and manufacturer of all materials used in this study are listed in Table (1).

LED (Bluephase C5, IvoclarVivadent] at 400 m W/ cm<sup>2</sup> was used in this study.

### Methods

#### Preparation of the composite resin specimens

A universal microhybrid commercial composite resin was used as control material and blended with the inorganic nanoparticles. A

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commercial universal nanofilled composite was used as a reference to compare with the nanoparticle-blended experimental composites (6,7).

Addition of CaCO<sub>3</sub> and ZnO nanoparticles

The CaCO<sub>3</sub> and ZnO nanoparticles treated with silane coupling agent were manually added to microhybrid RBCs in a dark room, at four different weight concentrations: 3% CaCO<sub>3</sub>, 5%CaCO<sub>3</sub>, 3% ZnO and 5% ZnO. The mixture will then thoroughly blended by speed mixture device (Karnavati, INDIA) in college of

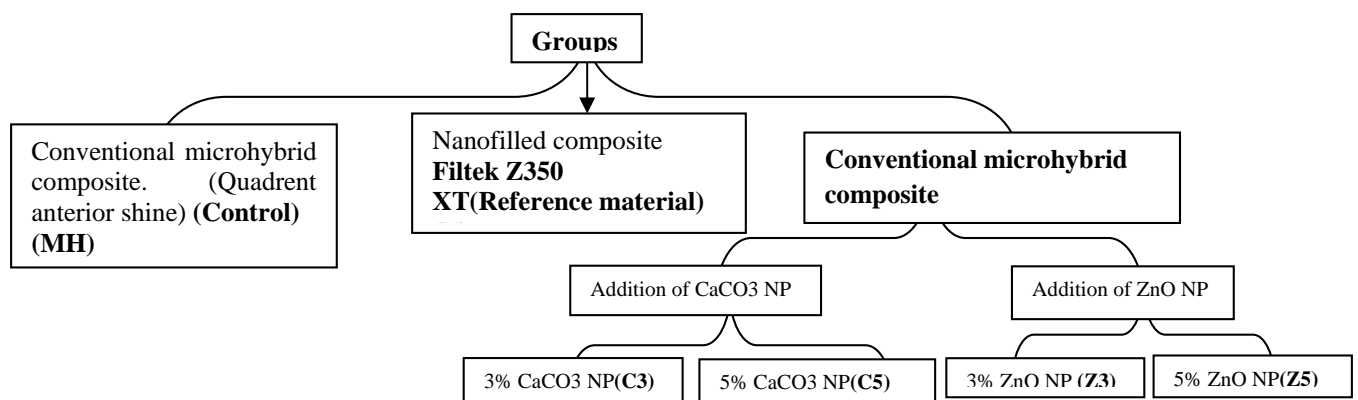
pharmacy/Hawler medical university. Before curing, the resulting paste packed into teflon molds using an oscillator to remove pores, and covered on both sides with a clear glass plate (6, 7, 8).

Groups design:

Six groups of samples denoted MH, N, C3, C5, Z3 and Z5 were defined. The nanoparticle type and weight ratio characterizing of each group are shown in figure 1. Each of these groups was subjected to radiopacity test evaluation.

**Table 1: The commercial name, the composition and manufacturer of the materials used**

Materials	Composition	Manufacturer
<b>Filtek Z350</b>	Bis-GMA, UDMA, TEGDMA,, Bis-EMA Fillers (78.5%W, 59.5% V): Combination of non- agglomerated/ non- aggregated 20 nm silica filler, non- agglomerated/ non- aggregated 4-11 nm zirconia filler, Aggregated zirconia/silica cluster filler.	3M ESPE, St Paul, MN, USA
<b>Quadrant Anterior Shine</b>	Bis-GMA, acrylates Fillers (75.6% W, 63% V) Barium glass, Silica, silicate glass, fluoride containing fillers (0.7 µm), Polymerization crystal, In-organic pigment	Cavex Holland BV, Haarlem, The Netherlands
<b>Zinc oxide nanofillers ZnO</b>	Nanofiller with (10-30 nm) coated with silane coupling agent (NH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> Si(OC <sub>2</sub> H <sub>5</sub> ) <sub>3</sub> )	SkyspringNanomaterials, Inc. USA
<b>Calcium carbonate nanofillers (CaCO<sub>3</sub>)</b>	Nanofillers (80 nm) coated with silane coupling agent	M K Impex Corp. Canada

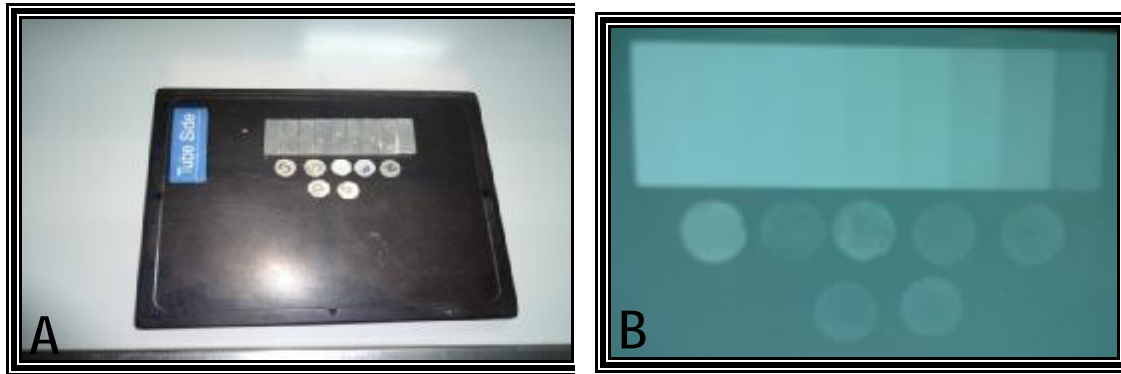


**Figure 1: Diagrammatic illustration of experimental design of groups for the study.**

Radiopacity evaluation

Five specimens for each group were prepared in the form of disks 15 mm in diameter and 1 mm thick. Each sample was placed on cassette film 20 × 25 cm in size with the Aluminum step-wedge as a standard to compare the radiodensity (Fig.2). The radiographic exposure was done using an X-ray unit machine (AXIOM, Iconos-R100, Siemens, Germany), operated for 0.5 s at 60 kV

and 1 mA The film-object distance was 40 cm. The radiographs film was developed and the optical density of radiographic film was analyzed with a transmission densitometer. The measured value was converted in terms of the equivalent thickness of aluminum by referring to the calibration curve for the radiographic density of an aluminum step-wedge (9).



**Figure 2:** (A) One specimen from each tested material, and Al step wedge positioned on film cassette. (B) Representative developed radiograph of the specimens and Al step wedge.

### Statistical Analysis

The means and standard deviations for optical densities (OD) of the specimens and aluminum step wedge of each radiograph were calculated by averaging the three repeated measurements to create a single value for each specimen. A linear regression analysis was calculated for each film, relating the OD of the steps in the wedge to the thickness of each step. The aluminum equivalent (Al) was then calculated for each sample by using the regression analysis equation of:

$$y = a + bx,$$

where:

y = the optical density (OD) of the specimen;

a = the coefficient of the regression;

b = the regression constant and

x = the aluminum equivalent value for that sample.

Descriptive analysis, One-way ANOVA and Duncan test were used to determine statistical significance of radiopacity among the materials.

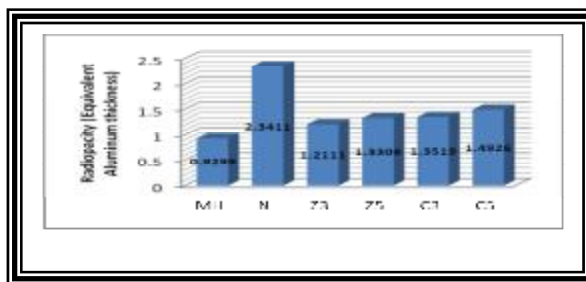
### RESULTS

The means and standard deviations of the radiopacity value of the tested materials are shown in table 2 and figure 3. According to the International Standards Organization (ISO) 4049, the radiopacity of a 1.0 mm thick composite specimen should be equal to or greater than the same thickness of aluminum. Only the MH group did not meet this criterion (0.9293 Al Eq.) which had the lowest radiopacity value and it is significantly different from the other groups. The addition of 3% of CaCO<sub>3</sub> and 3% ZnO to the microhybrid composite (MH) significantly increase its radiopacity. While the addition of 5% of CaCO<sub>3</sub> and 5% ZnO to the microhybrid composite (MH) increase its radiopacity but they were statistically not significant. The obtained result showed that the N group (nanohybrid composite resin) had the highest radiopacity (2.6242 Al Eq.) which is significantly different from the other groups.

**Table 2: Means and standard deviations of the radiopacity of the tested materials**

Groups	N	Mean	S.D.	S.E.	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
MH	5	0.929387 a	.0707107	.0316228	.841588	1.017186	.8294	1.0294
N	5	2.624296 d	.7071068	.3162278	1.746307	3.502285	1.6243	3.6243
Z3	5	1.773314 bc	.0642417	.0287298	1.693548	1.853081	1.6743	1.8543
Z5	5	1.0940 a	.0120181	.0053747	1.068366	1.098211	1.0706	1.0953
C3	5	1.566230 b	.1238230	.0553753	1.412484	1.719977	1.4944	1.7803
C5	5	1.083289 a	.0120181	.0053747	1.068366	1.098211	1.0706	1.0953
ZC	5	2.083573 c	.0378716	.0169367	2.036549	2.130597	2.0593	2.1457
Total	35	1.591911	.6322689	.1068730	1.374719	1.809103	.8294	3.6243

Note: Means with different letter indicated statistically significant ( $p < 0.05$ ).



**Figure 3: Bar chart for the means of radiopacity of tested materials**

## DISCUSSION

Radiopacity of a material can be simply defined as the inverse of the optical density of a radiographic image. Optical density value is a logarithmic measure of the ratio of the transmitted-to-incident light through the film image, measured by the transmission densitometry, it depend on the inherent X-ray absorption properties of the materials (10,11).

Radiopacity depends in part on selection of the polymer matrix, chemical nature of the filler particles, their size, density and an amount in the resin matrix, while resin matrices contribute little to the radiopacity of the material, it is typically the inorganic filler component that contributes most to the radiopacity of resin-based luting materials (11).

According to the International Standards Organization (ISO) 4049, the radiopacity of a 1.0 mm thick composite specimen should be equal to or greater than the same thickness of aluminum to be deemed radiopaque which is close to that of human dentin (3-5). In order to make comparisons between the different studies possible, aluminum step-wedge was chosen as a standard for measuring radiopacity, because its linear absorption coefficient ( $\mu$ ) is the same order as dental enamel (12,13).

According to the results of this study all the tested groups met the criteria of (ISO) 4049 except the MH group (0.9293 Al Eq.) which had the lowest radiopacity value and it is significantly different from the other groups. This might be due to the fact that this material contains SiO<sub>2</sub> (which are not radiopaque fillers) and a small percentage of Ba-F-Si fillers in its formulation (Table 1), in addition F and Si fillers had low atomic number which were: 9,14 respectively (4). This result agrees with the study of Sabbagh et al. (13). This can be explained in accordance with the manufacturer's propositions, that this composite material should be used only for anterior restorations.

On the other hand Filtek Z 350 XT composite resin (N group) showed the highest radiopacity value (2.6242 Al Eq.) which is significantly differ from the other groups, this is contributed to high fillers content of this composite and in addition to the presence of zirconium in its filler composition (table 1) which are radiopaque fillers and it's a high atomic number element (Zr=40).

C3 and Z3 groups showed significantly higher radiopacity value compared to MH group and they were not significantly differing from each other. This could be due to the fact that Zinc in ZnO nanoparticle had a high atomic number (Zn=30) and Calcium in CaCO<sub>3</sub> had a high atomic number (Ca= 20), while Z5 and C5 groups had lower RO value than Z3 and C3 with statistical significant difference. This might be attributed to the fact that incorporation of higher percentage (5%) of the particles into microhybrid composite act as light scatterers, hindering light penetration at depth, especially particles with a size that approaches the output wavelength of the light-curing unit.

Our result is agree with the study of Hewett et al. (15) who incorporated Calcium Carbonate fillers in different weight to make resin teeth more radiopaque, the result showed that the radiopacity increase by increasing the weight to 18gm then the RO value decline. Also agrees with the study of Moldovan et al. (16) who incorporated ZnO into composite resin in two different concentrations. The result showed that ZnO fillers have benefit effect on radiopacity, but the radiopacity was decreased as the concentration increased.

Finally variation in radiopacity measurements among different studies depends on a number of factors, including speed of the X-ray film, exposure time, voltage used and the age of the developing, fixing solutions, source-film distance, intensifying screens and specimen used (11).

As a conclusion; the addition of 3% of both the ZnO and CaCO<sub>3</sub> nanofillers fillers alone or in combination to microhybrid composite significantly increased the RO, while the addition of 5% of CaCO<sub>3</sub> and ZnO nanofillers to

microhybrid composite showed non-significant increase in the RO of the composite.

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