



# Influence of Bioactive Particles on Properties of Resin Infiltrant



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## Abstract

**Introduction:** This study is aimed to assess the effect of nanohydroxy apatite incorporation into the resin infiltrant on its properties.

**Materials and Methods:** The total sample size in the three groups was 60 specimens. The specimens were randomly divided in three main groups (ICON, ICON + 5% NHA, ICON + 10% NHA). Water sorption and solubility were evaluated as defined by ISO 4049 specification. The color stability specimens were evaluated with spectrophotometer. Color change was calculated to determine the extent of color stability between the tested groups.

**Results:** a statistically significant difference was observed among the tested groups relating to the water sorption, solubility and the color stability.

**Conclusion:** This study offers a promising route to greatly enhance resin infiltration treatment regarding its durability and color stability.

**Clinical Significance:** Integrating nanohydroxyl apatite particles in resin infiltrant is a promising route for treatment of dental caries.

## Introduction

Dental caries is considered one of the most prevailing diseases affect humans. The avoidance of dental caries and the remineralization of demineralized surface lesions before restorative interference is the goal of modern dentistry [1]. Enamel demineralization is the first sign of dental caries which jeopardizes tooth health and esthetics. Remineralization of these lesions is capable to arrest and reverse the lesion to healthy enamel tissue. The treatment of these lesions is usually performed with fluoride application. Even so, chronic low-level exposure to fluoride can result in some health problems (cancer, birth defects, bone health problems, gastrointestinal tract problems) additionally to dental fluorosis. For that reason, it is still essential to seek substitute, effective non-fluoride treatment that can afford an inclusive treatment for caries [2,3]. Following the up to date idea of minimal invasive dentistry, resin infiltration recently represented an unconventional approach to arrest demineralized lesions. The infiltration technique consists in using hydrophilic and high flowable, light-cured resins that can infiltrate subsurface micro pores, restraining the path of pathogenic bacteria and by-products and avoiding additional lesion development [4,5].

Previous studies have revealed that resin infiltration significantly enhances the micro hardness of initial demineralized

lesions. Lately, the advantages of the resin infiltration technique have been recorded: mechanical reinforcement of demineralized enamel, sealing of superficial porous and demineralized areas, and detain of lesion progress by increasing resistance to demineralization, minimized risk of recurrent caries and elevated patient acceptance [6-8]. Introduction of nanotechnology to the dentistry has dramatically changed the research areas. The nano-sized particles can be dispersed in higher filler concentration and polymerized into the resin system. Nano-hydroxy apatite (NHA) is considered one of the most biocompatible and bioactive material, and its nano sized particles are similar in morphology, crystal structure and crystalline of tooth enamel. NHA was reported to remineralize artificial caries lesion following its addition to toothpastes and mouth washes [9-12].

## Hypothesis

Incorporation of nanohydroxy apatite with resin infiltrant will have positive impact on its properties.

## Objectives

This study is aimed to assess the influence of nanohydroxy apatite incorporation into the resin infiltrant on its properties.

## Materials and Methods

### Sample size

The total sample size in the three groups (ICON, ICON + 5% NHA, ICON + 10% NHA) was estimated to be 60 specimens. The specimens were randomly allocated in three test groups.

### Specimens' preparation

Nano hydroxy apatite (NHA, (SIGMA-ALDRICH-USA) resin infiltration were prepared by adding Nano hydroxy apatite powder to the resin infiltration materials (ICON, DMG, Hamburg, Germany) in ratio of 5%, and 10% by weight using electric digital scale (AG245 Metter, Switzerland). The powder was mixed with resin using electric mixer.

- i. **In Group I:** resins infiltration (RI), without NHA,
- ii. **Group II:** 5% of NHA resin infiltration (5%NRI) while in,
- iii. **Group III:** 10% of NHA resin infiltration (10%NRI) were employed.

NRI were prepared by adding NHA powder to the resin infiltration materials (Icon) in ratio of 5%, and 10% by weight using electric digital scale. The entire materials were mixed by dual centrifugal laboratory vacuum mixer system (SpeedMixer.co.uk) for one min at speed 1300 rpm. Photo activation process for the resin were done by a light emitting diode (LED) light curing unit (LEdition, ivoclar vivadent, Austria). The curing was applied for 20 sec on each side, with the tip away of one mm.

### Water sorption (Wsp) and solubility (Wsl)

For specimen's preparation the resin was applied in a teflon mold with 12 mm diameter and 1mm thick. The mold was positioned between clear strips and glass slides under pressure, any excess material was removed. The cured specimens were stored in dark and dry container for 24 h, and afterward relocated to desiccators at  $37 \pm 1$  °C. After 22 h in the first desiccators, the specimens were transferred to second desiccators for 2 h at  $25 \pm 1$  °C, and then the weight of each specimen was recorded using an electric digital scale. In anticipation of a constant mass (m1) was gained (the variation was less than  $\pm 0.0001$  g) the cycle was repeated. Then the specimens were immediately immersed in deionized water at  $37 \pm 1$  °C, and measurements were obtained again after 60 days (m2). Following this step, the specimens were subjected to the previously mentioned process to gain a constant dry mass (m3), as the cycle described for m1. The values of (Wsp) and (Wsl) were calculated by the following equations as mentioned by ISO 4049 specification [13].

$$Wsp = 100 \times ((m2 - m1)/m1)$$

$$Wsl = 100 \times ((m1 - m3)/m1)$$

### Color stability

The oval disc shaped specimens were fabricated in teflon mold 12 X10 mm in diameter and 2 mm in thickness as required

by ISO International Standard #7491:2000 [14]. After curing, specimens were stored in dry and dark container at  $37$  °C for 24 h. Color readings were recorded 24 h after polymerization (baseline) and second reading after 60 days of immersion in distilled water at  $37$  °C in dark container. Water excess was removed from the specimens before readings by blotting papers. Water medium were changed weekly. Spectrophotometry (Color-Eye 7000A spectrophotometer, X-rite, Grand Rapids, Michigan, USA) was used to determine the color values consistent with The CIE Lab system (Commission International de l'Eclairage  $L^*a^*b^*$ ).

The original color measurement was recorded after 24 hours immersion in distilled water for each specimen as base line measurements. CIE-Lab is conveyed by the  $L^*$  coordinate, stand for color luminosity, varying from white to black; and the  $a^*$  and  $b^*$  coordinates, stand for the chromaticity of the color, with axes varying from green to red and blue to yellow, respectively. This color space is illustrated by a sphere, where the Y axis stands for the  $L^*$  parameter (lightness, from 0 = black, up to 100 = white), the X axis is the  $b^*$  parameter (from -b = blue, up to +b = yellow) and the Z axis represents the  $a^*$  parameter (from -a = green, up to +a = red), The match of these coordinate outcomes a spatial position that mathematically represents the color. All documented values were based on D65 illuminant and  $10^0$  observer geometry. For calibration, the spectrophotometer was adjusted over white and black backgrounds. Three measurements were done in the top surface of each specimen. The color changes ( $\Delta E$ ) were mathematically calculated by using the following formula [15].

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2};$$

$$\text{Where } \Delta L = L (60 \text{ days}) - L (\text{baseline}),$$

$$\Delta a = a (60 \text{ days}) - a (\text{baseline}),$$

And  $\Delta b = b (60 \text{ days}) - b (\text{baseline})$ ; where "baseline" values were obtained 24 h after the photo activation process.

### Statistical analysis

Numerical data were checked for normality by evaluating the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Water solubility and sorption data showed normal (parametric) distribution on the other hand color parameters data showed non-parametric distribution. Parametric data were presented as mean, standard deviation (SD), 95% Confidence Interval values while non-parametric data were presented as median and range values. For parametric data, one-way Analysis of Variance (ANOVA) was used to compare between the groups. Bonferroni's post-hoc test was used for pair-wise comparisons. For non-parametric data; Kruskal-Wallis test was used to compare between the three groups. Dunn's test was used for pair-wise comparisons. The significance level was considered at  $P \leq 0.05$ . Statistical analysis was performed with IBM (IBM Corporation, NY, USA), SPSS (SPSS, Inc., an IBM Company) Statistics Version 20 for Windows.

**Results**

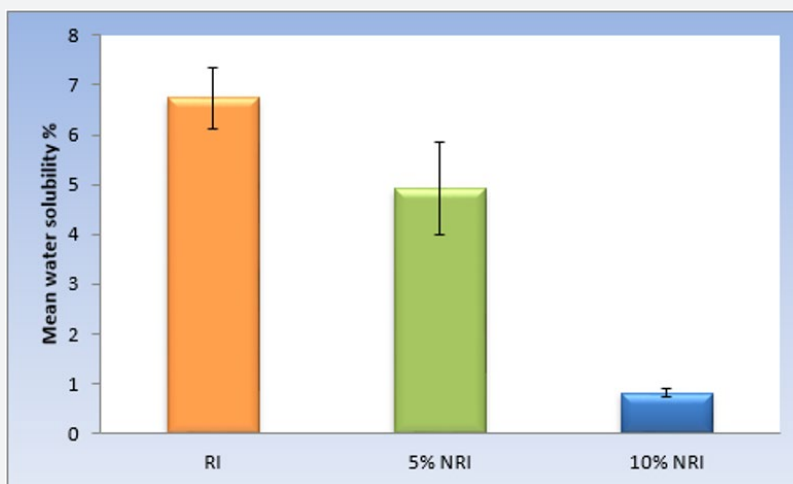
**Water solubility**

There was a statistically significant difference between the groups (P-value <0.001, Effect size = 0.943). Pair-wise comparisons

between the groups showed that RI group statistically scored the highest mean solubility. 5% NRI group showed lower mean value. While 10% NRI group showed statistically the lowest mean water solubility (Table 1 & Figure 1).

**Table 1:** Descriptive statistics and results of one-way ANOVA test for comparison between water solubility (%) of the three groups.

	RI	5% NRI	10% NRI	P-value	Effect Size (Eta Squared)
Mean (SD)	6.74 (0.61) <sup>A</sup>	4.92 (0.93) <sup>B</sup>	0.81 (0.08) <sup>C</sup>	<0.001*	0.943
95% CI	6.3 – 7.17	4.26 – 5.59	0.75 – 0.87		



**Figure 1:** Bar chart representing mean and standard deviation values for water solubility %.

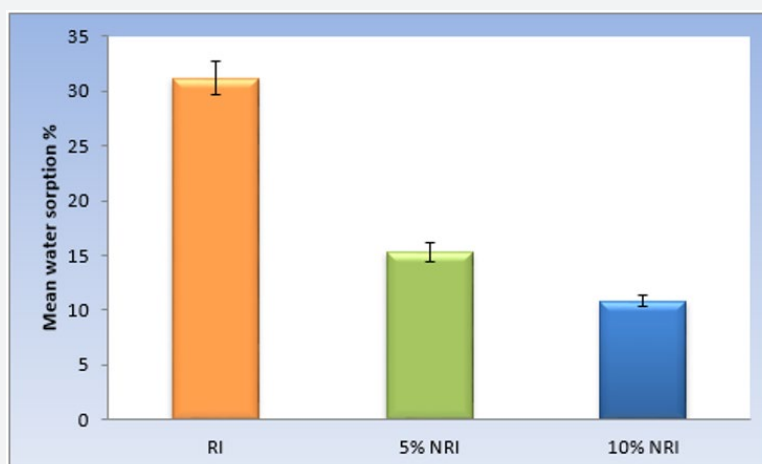
**Water sorption**

There was a statistically significant difference between the tested groups (P-value <0.001, Effect size = 0.987). Pair-wise

comparisons between the groups revealed that RI group showed statistically the highest mean sorption. 5% NRI group showed lower mean value. 10% NRI group showed the lowest mean water sorption (Table 2 & Figure 2).

**Table 2:** Descriptive statistics and results of one-way ANOVA test for comparison between water sorption (%) of the three groups.

	RI	5% NRI	10% NRI	P-value	Effect Size (Eta Squared)
Mean (SD)	31.17 (1.51) <sup>A</sup>	15.3 (0.86) <sup>B</sup>	10.81 (0.48) <sup>C</sup>	<0.001*	0.987
95% CI	30.09 – 32.24	14.68 – 15.91	10.47 – 11.15		



**Figure 2:** Bar chart representing mean and standard deviation values for water sorption %.

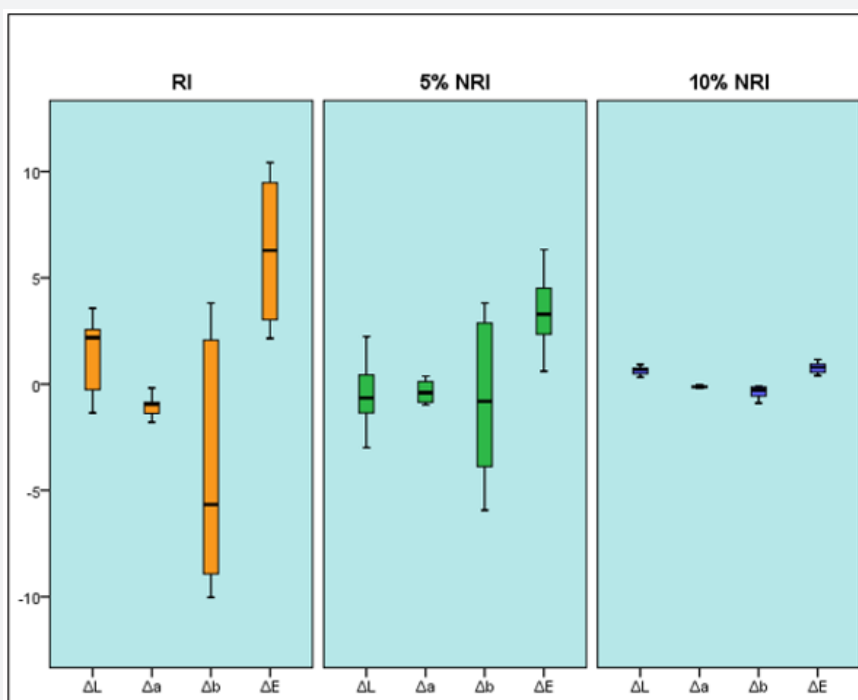
**Color parameters**

As regards  $\Delta L$ ; there was a statistically significant difference between the tested groups (P-value = 0.013, Effect size = 0.247). Pair-wise comparisons between the groups showed that RI group statistically recorded the highest median  $\Delta L$ . 10% NRI group scored lower median value. 5% NRI group was graded the statistically significantly lowest median  $\Delta L$ . As regards  $\Delta a$ ; there was a statistically significant difference between the tested groups (P-value <0.001, Effect size = 0.492). Pair-wise comparisons between the groups showed that RI group has the highest median

$\Delta L$ . 5% NRI group showed lower median value. 10% NRI group was the statistically significantly lowest median  $\Delta a$ . While for  $\Delta b$ ; no statistically significant difference between the groups was observed (P-value = 0.188, Effect size = 0.050). As regards  $\Delta E$ ; there was a statistically significant difference between the tested groups (P-value <0.001, Effect size = 0.616). Pair-wise comparisons between the groups showed that RI group has the highest median  $\Delta E$ . 5% NRI group showed lower median value. 10% NRI group has the statistically significantly lowest median  $\Delta E$  (Table 3, Figure 3).

**Table 3:** Median, range values and results of Kruskal-Wallis test for comparison between color parameters of the three groups.

	RI	5% NRI	10% NRI	P-value	Effect Size (Eta Squared)
$\Delta L$	2.18 (-1.35 – 3.56) <sup>A</sup>	-0.65 (-2.99 – 2.24) <sup>C</sup>	0.67 (0.32 – 0.92) <sup>B</sup>	0.013*	0.247
$\Delta a$	-0.97 (-1.79 – -0.19) <sup>A</sup>	-0.41 (-0.98 – 0.37) <sup>B</sup>	-0.12 (-0.21 – -0.03) <sup>C</sup>	<0.001*	0.492
$\Delta b$	-5.67 (-10.03 – 3.81)	-0.82 (-5.93 – 3.81)	-0.28 (-0.89 – -0.08)	0.188	0.05
$\Delta E$	6.28 (2.15 – 10.41) <sup>A</sup>	3.29 (0.61 – 6.32) <sup>B</sup>	0.79 (0.39 – 1.15) <sup>C</sup>	<0.001*	0.616



**Figure 3:** Box plot representing median and range values for color parameters in the three groups.

**Discussion**

The most significant properties determining the durability and longevity of dental restorations in the oral cavity is dimensional stability against dissolution and disintegration. The most common laboratory test of solubility is the ADA specification No. 8 (American Dental Association) which was followed in this study. Resin monomers are the principal chemical components of composite restorative materials. Both acrylates and methacrylates monomers are susceptible to water degradation (hydrolysis) of their ester group. Due to its hydrophilicity it may lead to water

uptake, which primes to higher hydrolysis susceptibility of the monomers along with discoloration of the cured resin [16]. Although TEGDMA revealed the best ability to infiltrate deep into the demineralized caries lesion [6]. It has been reported that, TEGDMA has the most water sorption affinity, followed by Bis GMA and by UDMA. Thus, in this study TEGDMA resin monomer is the main compositions of the resin infiltration. This may explain the tendency of the icon to water sorption and solubility. Additionally, the resin infiltration Icon does not include any filler leads to its higher susceptibility to water sorption and solubility than the groups with NHA fillers [17].

Maintaining color stability in the oral cavity is a fundamental property for tooth colored restorative materials. Even though improvements have been accomplished recently, discoloration is still a problem [18]. Many researches approved the effectiveness of the infiltration treatment technique with Icon of non-cavitated caries lesions [4,5]. Though, maintaining color stability of the resin over time is mandatory [19]. The method in the present study was in accord with several researches that used spectrophotometry and the CIE L\*a\*b\* coordinate system, which is a widely used tool for dental purposes. Many studies recorded the advantages of the CIE L\*a\*b\* coordinate system, for instance its repeatability, sensitivity, and objectivity [19]. This technique was preferred in evaluation the color change ( $\Delta E$ ) because its ability for the detection of small color variations [20].  $\Delta E$  values between 1 and 3 are perceptible to the naked eye and  $\Delta E$  values greater than 3.3 are considered clinically unacceptable [21-23].

In this study, although all the materials tested have demonstrated color changes after the immersion time; some variances have revealed between the tested materials. Icon recorded significantly higher variation in  $\Delta L$  (lightness) and  $\Delta a$  (green-red component) values. Regarding the total color changes ( $\Delta E$ ), Icon also scored the highest values ( $\Delta E$  6.28) which can be considered clinical unacceptable. This may be explained by a breakdown of the matrix components overtime degradation of the resin coupled with water sorption of TEGDMA, which leads to resin discoloration [24]. Hence, in this study Icon (composed mainly of TEGDMA and not include any filler) became more discolored as compared with other groups. Our results are in accordance with recent studies, which observed that Icon presents significant alteration of color after staining procedures [25,26]. On the other hand, clinical studies, concluded the acceptable color stability of resin infiltration in the oral environment, These Authors revealed that the color stability can be achieved by polishing to remove any stain [27,28]. In the present study, a long-term exposure with specimens stored for 60 days in distilled water was performed, which is considered sufficient for long-term staining ability evaluation [29].

### Conclusion

Results concluded from the present study could be of clinical relevance. It is contemplated that while Icon can enhance the esthetic problem associated with demineralization, the resin may discolor over time, nevertheless, this has been investigated on pure Icon discs without the underlying tooth structures. Incorporation of NHA with Icon will enhance its color stability and decrease its water sorption and solubility.

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