ORIGINAL RESEARCH

Comparative Evaluation of Solubilities of Two Nanohybrid Composite Resins in Saliva Substitute and Distilled Water: An *in vitro* Study

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ABSTRACT

Aim of the study: To determine the solubility of nanohybrid (NH) composite resins when immersed in saliva substitute and distilled water.

Materials and methods: A total of 60 disc-shaped samples (n = 30) were prepared in teflon molds of diameter 10 mm × 2 mm thickness from two NH composite resins. They were light-cured following which the samples were removed and weighed three times using a digital balance. Samples were then immersed in test solutions, i.e., 100 mL of distilled water and saliva substitute each stored for 24 hours and 7, 14, 21, and 28 days in an incubator at 37°C at 100% humidity. At the time intervals mentioned, the samples were removed from the incubator, washed with distilled water, blotted dry, and weighed. Samples were again immersed in fresh test solutions. Difference in preweight and postweight of samples was calculated and data were analyzed using appropriate test by testing normality. A p-value < 0.05 was considered to be significant.

Results: The results were analyzed using one-way analysis of variance (ANOVA). As observed from the baseline weight to the 28th day weight, there was a loss of weight of the samples in all the groups except NH composite resin (Flash) in distilled water.

Conclusion: From the results of the study, it was observed that there was no statistically significant difference in weight, with respect to the solubility of the two NH composite resins and the two solutions tested. Clinical significance of the study is that the NH composite resin that shows lesser solubility can be the choice for an esthetic restorative material in patients for long-term results.

Keywords: Artificial saliva, Distilled water, Nanohybrid composite resin, Solubility.

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INTRODUCTION

Composite resins have been widely used since their introduction because they possess excellent esthetic properties.[1] Currently, available dental composites resins provide satisfactory strength and high esthetic appearance. With the introduction of nanotechnology, the so-called nanocomposites with improved and favorable mechanical properties are available to the clinicians.[2] Thus, NH composite resins have been developed and marketed during recent years. The novel technique of nanotechnology has enabled incorporation of nanofiller particles in the organic resin matrix of dental composites at a nanoscale, resulting in a substantial improvement in mechanical, esthetic, and optical properties as compared with those of the conventional, microfilled, and hybrid resins.[3] One of the problems associated with these restorative materials is solubility as they are continuously bathed in saliva. Solubility of NH composite materials is of great importance in restorative dentistry, since inorganic ions present as fillers within composites can leach into the surrounding environment resulting in breakdown of the restoration.[4] Breaking of chemical bonds in the resins or softening through the plasticizing action of water are the results of the hydrophilic degradation.[5] In spite of highly cross-linking networks in polymerized composites, a few other components may be eluted into water, such as residual monomers, small-chain polymers, and polymerization promoters and ions from filler particles. Most of these leachable species are eluted quickly from polymerized resins within a few days. Consequently, the esthetic performance and biocompatibility of the materials may be compromised by the release of these components.[6] Previous studies have established that water sorption in dental resin composites is a diffusion-controlled process.[7,8] It has been observed that a number of locally manufactured composite resins are available to the clinician, but their performance as compared with the gold standard available materials remains to be tested. Thus, the aim of this study was to evaluate and compare the solubilities of two NH composite resins (standard and locally manufactured) in artificial saliva and distilled water.

Table 1: Composition of nanohybrid composite resins used

Composite resin	Oraganic/inorganic matrix	Filler	Filler size	Shade
Tetric N Ceram group NH1	Bisphenol A-glycidyl methacrylate (BISGMA), urethane dimethacrylate (UDMA) or (semicrystalline polyceram) (PEX), and an inorganic filler, such as silicon dioxide (silica)	An inorganic filler, such as silicon dioxide (silica)	0.6 μm	A2
Flash group NH2	Bisphenol A-glycidyl methacrylate (BISGMA), urethane dimethacrylate (UDMA) or (semicrystalline polyceram) (PEX), and an inorganic filler, such as silicon dioxide (silica)	Inorganic filler, such as silicon dioxide (silica)	0.4 μm	A2

MATERIALS AND METHODS

Two NH composite resin materials [one standard: Tetric N Ceram (Ivoclar Vivadent India) and the other locally available: Flash (Medicept Dental, India)] were selected for this study, and the solubilities of the materials were tested in artificial saliva and distilled water. The composition of the two NH composite resins tested is as shown in Table 1. For preparation of samples, a clean glass slab was kept on a flat surface and a Teflon mold was placed over it for the fabrication of the samples. The NH composite resin was dispensed and placed into the mold with the help of composite filling instrument. A Mylar strip was placed beneath and over the composite resin. A microscopic glass plate was used to compress the NH composite resin on top to remove the excess and to flatten and smoothen the surface. The composite resin in the mold was then light-cured from both sides as per manufacturer's instruction using a halogen light-curing unit (Guilin Woodpecker Medical Instrument Co Ltd) (Fig. 1). Following this, the samples were removed from the mold and polished using composite polishing kit (Shofu Dental Corporation, Japan). Likewise, 60 samples measuring 10 mm × 2 mm dimension were prepared from both NH composite resins (30 in each group: NH1 and NH2) of shade A2 enamel. These composite resin samples were kept in the dark for 24 hours, so that complete polymerization occurs. After 24 hours, all the samples were preweighed up to four decimal points using a digital weighing balance (Contect India, Turbhe, Navi Mumbai). Following this, the two groups NH1 (30 samples) and NH2 (30 samples) were further divided into two subgroups of 15 each to be immersed in artificial saliva and distilled water. They were stored in labeled 10-mL bottles filled with artificial saliva and distilled water in the dark
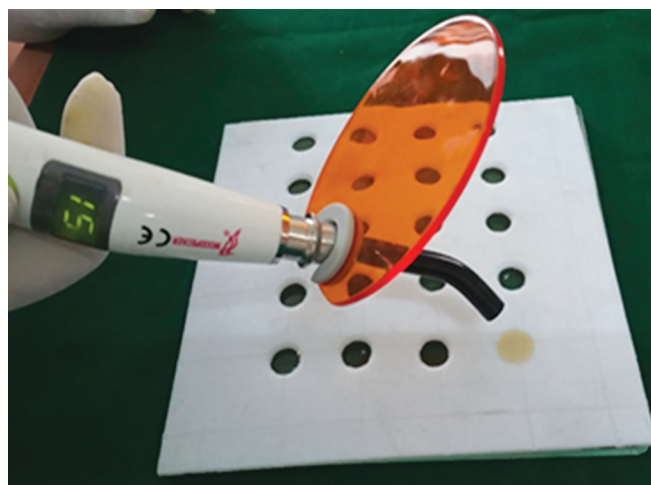

Fig. 1: Sample preparation using Teflon molds

at 37°C in an incubator. The samples were removed from test solutions, blotted dry, and weighed with the digital weighing balance at 24 hours and 7, 14, 21, and 28 days intervals. After each interval, they were immersed in fresh test solutions and stored again in the dark at 37°C in an incubator. The data obtained were analyzed statistically using ANOVA test. A p-value < 0.05 was considered to be significant.

RESULTS

The NH composite resin (Tetric N Ceram) showed a decrease in weight from baseline up to 14 days and thereon at the end of the 21st day, it was observed to have gained weight, but at the end of the test period of 28 days, it was observed to have lost weight in artificial saliva. There was, however, a steady decrease of weight in distilled water at the end of the observation period (Table 2 and Graph 1). The NH composite resin (Flash) showed a

Table 2: Multiple comparisons of ANOVA test to find out which pairs differed significantly

Time	Tetric N Ceram in artificial saliva	Tetric N Ceram in distilled water	Flash in artificial saliva	Flash in distilled water
Baseline	0.4277 ± 0.05250	0.4388 ± 0.04388	0.3955 ± 0.04781	0.4179 ± 0.02538
24 hours	0.4277 ± 0.05250	0.4396 ± 0.04454	0.3957 ± 0.04767	0.4180 ± 0.02549
7 days	0.4264 ± 0.05222	0.4406 ± 0.04438	0.3957 ± 0.04800	0.4177 ± 0.02542
14 days	0.4257 ± 0.05228	0.4392 ± 0.04452	0.3949 ± 0.04792	0.4174 ± 0.02530
21 days	0.4272 ± 0.04977	0.4384 ± 0.04439	0.3946 ± 0.04796	0.4202 ± 0.02477
28 days	0.4243 ± 0.05210	0.4374 ± 0.04466	0.3942 ± 0.04820	0.4197 ± 0.02453
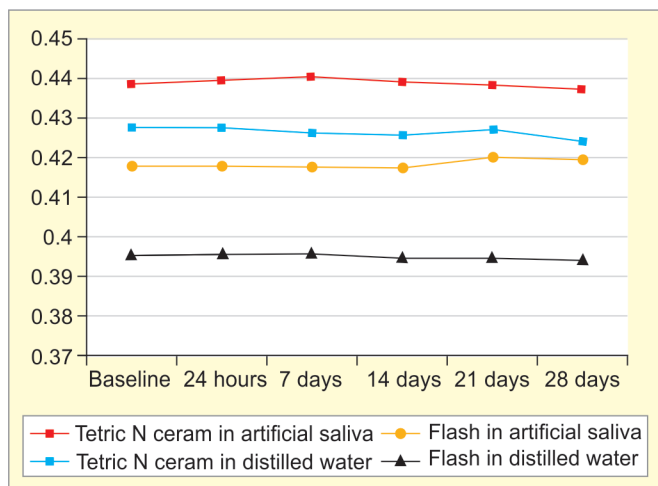
Graph 1: Comparison between the solubility of the two composite resins in artificial saliva and distilled water at baseline, 24 hours and 7, 14, 21, and 28 days. (X axis represents time interval in days and Y axis represents solubility in mm)

steady increase in weight from baseline to the end of the observation period of 7 days in artificial saliva and then a steady decrease in weight up to the 28th day. In distilled water, it showed an increase in weight at the end of 24 hours and then after a steady decrease in weight until the end of the 14th day period and gained weight. At the end of the 21st day, and decrease in weight at the end of 28th day (Table 2 and Graph 1). One-way ANOVA showed that water absorption was different for the tested materials. Flash in artificial saliva lost approximately twice the weight in μgm of Tetric N Ceram; however, Flash in distilled water showed an increase in weight at the end of the test period as compared with the initial weight (Table 2). Table 3 represents multiple comparisons of ANOVA test (p < 0.005).

DISCUSSION

Restorations in the oral cavity constantly come in contact with oral fluid and food. Composite resins always remain soaked in saliva in the oral cavity, and these NH composite resins may undergo water sorption and solubility, which may lead to degradation of the resin matrix and debonding of the matrix-filler interface, resulting in a deterioration of mechanical properties.[9] In addition,

Table 3: Multiple comparisons of ANOVA test (p < 0.005)

Groups	f-value	p-value	Significance
Tetric N Ceram + artificial saliva	0.010	1.000	NS
Tetric N Ceram + distilled water	0.009	1.000	NS
Flash + artificial saliva	0.002	1.000	NS
Flash + distilled water	0.032	0.999	NS
NS: Nonsignificant			

leakage of fillers, ions, and organic substances, such as residual monomers, methacrylic acid, and formaldehyde from resin composite material can occur as a result of exposure to an aqueous environment. Some of these organic substances can act as irritants and may induce delayed allergic reactions.[10] In the present study, it was observed that there was significant difference observed numerically in the baseline weight up to the 28th day of the NH Tetric N Ceram in artificial saliva and distilled water and the NH Flash in artificial saliva indicating the loss of weight of the composite resins, suggesting leaching of monomers from the samples. But, there was weight gain observed in the baseline and 28th day weight of the NH Flash in distilled water. Also, the two NH composites Tetric N Ceram, a standard available, and Flash, a locally manufactured composite, showed solubility in both the test solutions, i.e., artificial saliva and distilled water. The composition and the filler particle size of both the composite resins tested were similar though manufactured by different companies (Table 1). The results of our study demonstrated that there was no statistically significant difference in the weight of the samples at all the time intervals as compared with baseline, thus indicating no effect of the test solutions on the solubility of both NH composites except for Flash in distilled water (Table 3). The American Dental Association Specification No. 27 requires that "the solubility of all materials be less than or equal to 7.5 μg/mm^3 within a 7-day period of water storage."[11] When resin samples are immersed in water, some of the components, such as unreacted monomers or filler dissolve and are leached out of the samples. This results in loss of weight and can be measured as solubility or leaching.[12] A study done by Biradar et al[13] showed the maximum amount of water absorption in the first week, then a gradual decrease in the water absorption from the second to the 6th week, as compared with the 1st week. There was no statistically significant difference among the three tested composite resins. In another study done by Al-Shekhli and Al-Khfaji,[14] microhybrid and nonohybrid composite resins were compared with regard to their solubility in water. All the composites tested in the present study exhibited sorption and solubility values within the acceptable limits. Berger et al[15] in this study of water sorption and solubility of one NH and two microhybrid composite resins produced results that show that the NH composite resin showed lower solubility and sorption as compared with other two microhybrid composite resins. Several factors, such as the polymeric matrix composition, filler particle type and content, and the degree of curing reached after the polymerization reaction can influence the solubility of dental resin composites. As in this study, Tetric N Ceram and Flash had filler particle sizes of 0.6 and 0.4 μm, both showed

similar solubilities.[15] The test solutions used in this study were artificial saliva and distilled water. Saliva in the oral cavity contains 99.5% water. A aaliva substitute, which contains sodium carboxy methyl cellulose 0.5% w/v and glycerine 30%, resembles the composition of natural saliva and was used as an substitute for saliva present in oral cavity to simulate clinical conditions.[16] Teflon mold was used in this study for fabrication of samples as they do not adhere to the mold and can be easily prepared. The quartz–tungsten–halogen light-curing unit was used in this study, which had a tip diameter of 12 mm, intensity of 450 mW/cm^2, and wavelength between 400 and 500 nm, which is sufficient to cure composite resin restorations directly up to a depth of 2 mm. Specimen diameter was 10 mm, which was less than the tip diameter of light-curing unit (12 mm) to ensure complete surface of sample gets cured.[17] Mylar strip was kept above and below the samples during fabrication, so that the composite resin is confined inside the mold and also a flattened surface is obtained.[18] Glass plate used in the study was to compress, flatten, and smoothen the surface. Yet, the samples were polished to remove irregularities and give a final finish to the samples. Samples were placed in an incubator during the course of the study to facilitate a constant environment at 37°C and in the dark. This being a pilot study, the time interval studied was only for 28 days. Further long-term studies with increased sample size are required to observe the effects of artificial saliva on various composite resins available in the market.

CONCLUSION

The solubility of NH composites resins Tetric N Ceram and Flash in artificial saliva and distilled water used in this study showed similar solubility characteristics in both artificial saliva and distilled water solutions. There was no statistically significant difference in the solubilities of the composite resins when observed up to the 28th day.

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