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Research Report

Differences in surface roughness of nanohybrid composites immersed in varying concentrations of citric acid

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ABSTRACT

Background: *The surface roughness of restoration is important in predicting the length of time it might remain in the mouth. Conditions within the oral cavity can affect the surface roughness of a restoration. Nanohybrid composite is widely used in dentistry because it can be applied to restore anterior and posterior teeth. Athletes routinely consume isotonic drinks which are acidic and even more erosive than the carbonated variety because they contain a range of acids; the highest content of which being citric acid.* ***Purpose:*** *The aim of the study was to analyze the surface roughness of nanohybrid composite after having been subjected to immersion in varying concentrations of citric acid.* ***Methods:*** *Two isotonic drinks (Pocari Sweat and Mizone) were analyzed using high performance liquid chromatography (HPLC) to quantify the respective concentrations of citric acid which they contained. A total of 27 samples of cylindrical nanohybrid composite were prepared before being divided into three groups. In Group 1, samples were immersed in citric acid solution derived from Pocari Sweat. Those of Group 2 were immersed in citric acid solution derived from Mizone; while Group 3, samples were immersed in distilled water as a control. All samples were immersed for 7 days, before their surface roughness was tested by means of a surface roughness tester (Mitutoyo SJ-201). Data was analyzed using a one-way ANOVA test.* ***Results:*** *The results showed that there was no significant difference in surface roughness between Groups 1, 2 and 3 (p=0.985).* ***Conclusion:*** *No difference in surface roughness of nanohybrid composites results from prolonged immersion in varying concentrations of citric acid*

Keywords: *composite; nanohybrid; citric acid; surface roughness; pH*

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INTRODUCTION

Nanohybrid composite consists of both micrometer-sized and nanometer-sized particles[1] and represents a universal composite for both anterior and posterior restoration. It can also be used for esthetic purposes, being suitable for filling both posterior teeth requiring great pressure and anterior teeth.[2]

If the composite resin is eroded, teeth with fillings formed from this substance can suffer loss of anatomy and secondary caries, in addition to experiencing increased surface roughness of the restoration. This can, in turn, lead to the formation of plaque and deposits staining the restoration. This condition leads to irritated soft tissues which can develop into gingivitis, as well as a decrease in restorative resilience.[3] The surface roughness of composite resin is affected by filler content, volume, matrix type, and coupling agent disintegration within the surfaces of the composite resin fillers.[4]

Athletes consume isotonic drinks before, during, and after exercise to minimize dehydration and excessive changes in electrolyte balance.[5] In a number of countries, researchers have published data on the relationship between athletes and dental erosion. In the United States, some have reported that more than 35% of athletes experience tooth erosion.[6,7] A similar prevalence occurred in the United Kingdom, where 36-85% of athletes surveyed suffered from the condition.[8] It was also found to afflict 45% of athletes who participated in the London Olympic Games in 2012.[9] Dental erosion affected 25% of the 12-17 year old swimmers in Lithuania and 50% of 18-25 year old swimmers in Lithuania.[10] Similarly, an Australian study

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reported that between 20-30% of athletes surveyed suffered from dental erosion.[11]

One of the factors affecting dental health is the consumption of isotonic drinks with low pH, i.e. approximately pH 2.4 to 4.5, which is below the critical pH level. The majority of soft drinks, including the isotonic variety, contain several types of acids, such as phosphoric, citric, malic, and tartaric.[12,13] Previous research also reported that energy drinks consumed during exercise contain citric acid which damages the organic fillers of composite.[14] In fact, citric acid is a weak acid often used as an additive in food and drinks. Furthermore, there is no clear-cut critical pH concentration below which erosion will occur.[15] Therefore, this study aimed to analyze the surface roughness of nanohybrid composites immersed in varying concentrations of citric acid.

MATERIALS AND METHODS

This laboratory-based experimental research employed a post test-only control group design. The concentrations of citric acid solution used were adjusted to those contained in some isotonic drinks available in Indonesia. Isotonic drinks analysed in this research were Pocari Sweat and Mizone, widely consumed by the public in Indonesia based on the Top Brand Index in the year 2011 and 2015. A high performance liquid chromatography (HPLC) test was performed in advance to determine the concentration of citric acid contained in each of the isotonic drinks. Based on HPLC test, the concentration in Pocari Sweat was 2509.2 ppm, while that of Mizone was 1897.6 ppm. Pure citriid acid was made for each test results in accordance to its concentration: pure citric acid with concentration of 2509.2 ppm has a pH of 2, while the one with a concentration of 1897.6 ppm has a pH of 3..

The research sample used nanohybrid composite resin (Filltek Z250 XT, 3M ESPE, MN, USA) in a cylindrical shape with a diameter of 5 mm and a thickness of 2 mm, activated by light curing method.[16] The number of samples was 27, divided into three groups. Group 1 used nanohybrid composites immersed in citric acid at a concentration of 2509.2 ppm. Group 2 used nanohybrid composites immersed in citric acid at a concentration of 1897.6 ppm. Meanwhile, Group 3 used nanohybrid composites immersed in distilled water as a control.

A cast for the samples was made of 2 mm thick acrylic plates and a hole diameter of 5 mm.[16,17] The cast was smeared with Vaseline and subsequently placed on a glass plate with a celluloid strip attached. Nanohybrid composite resin was then introduced into the cast on the glass plate covered with the celluloid strip and subjected to a weight load for 30 seconds to produce a flat and smooth surface.[18] The scales and glass plate were then lifted. Thereafter, polymerization was carried out using light curing units (Cure Rite, Caulk, Dentsply, Canada) at a wavelength of 400-500 nm and an average light intensity of 637 mW/cm^2 for 20 seconds (in accordance with the manufacturer's instructions). Sample preparation was conducted to obtain 27 samples with flat, smooth, shiny surfaces. The entire sample was randomly divided into three groups and then subjected to prolonged immersion in the solutions.

The samples were immersed for 7 days in each of the test solutions which were replaced daily in order to maintain their stability. The immersion time was determined based on the assumptions that, with each instance of drinking, residual beverage may remain in the mouth for about 15 minutes, and that the 7-day immersion is equivalent to a period of 672 days (7 x 24 hours x 60 minutes divided by 15 minutes per day) or about 2 years of isotonic drink exposure to restorative material in the oral cavity.[17] Immersion was conducted within sealed bottles placed in a sealed box at room temperature to avoid sunlight possibly negatively affecting the stability of the solutions.

After 7 days of immersion, samples were taken from each test group and then dried with blotting paper. The surface roughness of the samples was then investigated using a surface roughness tester (Mitutoyo SJ-201, California, America) with an accuracy of 0.01-100 µm. The parameter used in the surface roughness test was that of roughness average (Ra) which shows an average value for the surface roughness of the whole formation of the peaks and valleys recorded by the tool.[18,19] Data was collected twice from the intersection at the mid point of the samples.

The data obtained was tabulated for each group and analyzed for normality with a Kolmogorov Smirnov Test. A Levene test was used to measure the homogeneity of the data. Normally distributed and homogeneous data would be examined using a one-way ANOVA test to reveal any significant differences between the sample groups.

Table 1. Results of the difference test on the surface roughness of nanohybrid composites immersed in citric acid solution at different concentrations for seven days

Group	n	Mean (µm)	Standard Deviation (µm)	p
1	9	0.2211	0.05413	0.985
2	9	0.2183	0.04465	
3	9	0.2178	0.02682	

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RESULTS

This research focused on the surface roughness of nanohybrid composites immersed in citric acid solution at different concentrations. The results are shown in Table 1 and Figure 1. From the table and figure above, Group 1 composites immersed in citric acid at a concentration of 2509.2 ppm, had a similar mean surface roughness to that of Group 2 composites immersed in citric acid at a concentration of 1897.6 ppm. The mean surface roughness in Group 1 and Group 2 was 0.2211 and 0.2183 µm, respectively; while in group 3, composites immersed in distilled water as a control produced results of 0.2178 µm. These three findings were compared and tested statistically using one-way ANOVA, producing the result $p = 0.985$ ($p>0.05$). The result confirmed that there was no significant difference in the surface roughness of nanohybrid composites immersed in citric acid solutions of different concentrations.

DISCUSSION

Based on the results of this research, no differences existed in the surface roughness of the composites after immersion in pure citric acid solutions at certain concentrations equivalent to those of specific isotonic drink products in Indonesia, namely; Pocari Sweat and Mizone. The results of the HPLC test also indicated that the highest level of acid contained in those isotonic drinks was citric acid. Previous research had similarly found that citric acid is the most acidic ingredient in energy drinks and sport drinks.[14]

Such research had also used the same detector, a surface roughness tester, to detect the surface roughness of composites with the same accuracy value.[3,17] Nevertheless, this surface roughness tester (Mitutoyo SJ-201, California, America) has some limitations when used to examine the surface roughness of materials with nanometer-sized particles since it can only detect the loss of micrometer-sized composite particles (0.01-100 µm). Consequently, if the size of composite particles released is smaller, they will remain undetected.

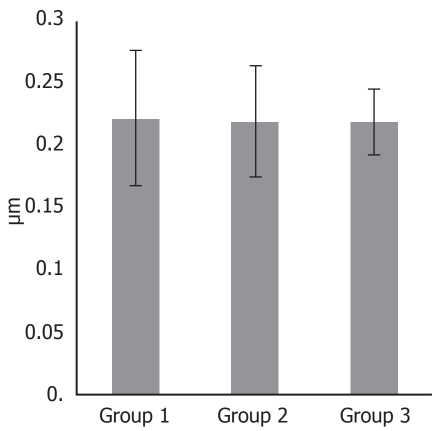

Figure 1. Mean surface roughness of nanohybrid composites immersed in citric acid solution at different concentrations for seven days.

The complex structure of a surface cannot be fully characterized by means of surface-only roughness measurements.[20] More valid predictions of clinical performance can be made when the surface roughness measurements are combined with an SEM analysis that permits evaluation.[20] A study by Ergücü *et al*., used roughness measurement and SEM to reveal the damage to the surface of all the resin composite tested. It was observed that roughness measurements were largely confirmed by SEM analysis.[20]

In this research, the results showed that citric acid alone did not cause the surface roughness of the nanohybrid composites. Citric acid is a weak organic acid, commonly used as a food additive. Therefore, the strength of the acid is insufficient to damage the bonds contained in the composites that have been perfectly polymerized.[15] The composites used in this research were nanohybrid in nature, which featured a combination of filler particle sizes, thus causing the bonding between fillers and matrix to be stronger. Nanohybrid composites also have high filler contents, so they have a higher resistance to acid conditions.[21] The strength of the acid that can cause erosion is not only dependent on the concentration or the pH of the solution, but also on the amount of acid that is available (titratable acidity), the degree of dissociation (pKA), and the function of acid as a chelating agent.[22]

There are some researchers who claim that soft drinks have a low pH and high titratable acidity (TA).[7,23] However TA is more important in determining the erosive potential of a drink rather than its degree of acidity (pH).[24] The degree of acidity (pH) merely represents the hydrogen ion concentration of a drink, while TA measures the total acid concentration of a solution.[25] Although the types of beverages are acidic, their pH values and their TA are different.[23] Based on the results of this research, there was no difference in the surface roughness of the composites between the group using citric acid solution with an acidity degree of 2 and the control group. In contrast to this research, a previous investigation using a drink with an acidity degree of 2.97 showed a difference in the surface roughness of nanohybrid composites.[3] Another previous piece of research showed that citric acid can dissolve or erode the surface of a tooth[15] and that citric acid has the capacity to chelate (chelating agent) so that it can interact with saliva as well as instantly soften and dissolve tooth mineral.[26] In this research, however, the test of the citric acid exposure was only conducted on the composite filling materials, which did not contain calcium ions. Moreover, it did not use saliva that contains calcium minerals, but only used distilled water. As a result, the role of citric acid as a chelating agent, which was thought to affect the potential for erosion, could not be detected. It can be concluded that there is no significant difference in the surface roughness

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of nanohybrid composites immersed in citric acid solution at different concentrations. Nevertheless, further studies into the surface roughness are expected to use another additional tool to detect the surface topography such as TA as a parameter substituting pH.

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