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RESEARCH ARTICLE

Surface roughness and colour changes of nanofilled composite resin after immersion in yogurt drink

Sindy Windya Ningrum*, Purwanto Agustiono**, Harsini**⊠

- *Master of Dental Sciences, Faculty of Dentistry, Universitas Gadjah Mada, Yogyakarta, Indonesia
- **Department of Biomaterial, Faculty of Dentistry, Universitas Gadjah Mada, Yogyakarta, Indonesia
- **Jl Denta No 1, Sekip Utara, Yogyakarta, Indonesia; ⊠ correspondence: harsiniugm@yahoo.com

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ABSTRACT

Nanofilled composite resin is one of restorative materials with some weaknesses, such as changes of colour and surface roughness. These weaknesses are attributed to some factors, including frequent consumption some beverages, including yoghurt drinks. This study aims to determine the effect of long immersion of nanofilled composite resin in guava yogurt drink on discoloration and changes in surface roughness. Subjects of the study were nanofilled composite resin materials (3M Filtek Z350XT shade A3) in cylindrical shape with a diameter of 10 mm and 2 mm thickness. Study was conducted by immersing nanofilled resin composites in 10 mL of guava yogurt drink at 37 °C. Twelve specimens were divided into 3 groups with immersion duration of 12 hours, 24 hours, and 36 hours. Surface roughness measurements were carried out using a profilometer Starrett SR300 surface roughness tester, while the colour measurement parameters according to the system L * a * b * was measured using chromameter Konica Minolta CR-400 before and after treatment. Data of changes in surface roughness and colour changes data (Δ E) were calculated and analysed using one-way ANOVA. The results showed insignificant surface roughness changes but significant colour changes in nanofilled composite resin in the groups of 12 hours, 24 hours, and 36 hours. It is concluded that immersion duration in guava yoghurt drink significantly affects the colour of nanofilled composite resin.

Keywords: colour changes; immersion; nanofilled; surface roughness; yogurt drink

INTRODUCTION

Composite resin is restorative material composed of three major components, such as matrix, filler, and coupling agent. Composite resin becomes the most popular dental restoration material because of its aesthetic properties. Depending on its filler, particle size composite can be differentiated to macrofilled (10-100 μm), small/fine fillers (0.1-10 μm), midfilled (1-10 μm), minifilled (0.1-1 μm), microfilled (0.01-0.1 μm), and nanofilled (0.005-0.1 μm). Nanofilled composite resin provides more advantages in terms of strength, modulus and polishing as compared to other composites. Composite resin can change in colours and roughness on surfaces such as other types of resins. 2,3

Indonesians are known to consume some favourite instant drinks, such as coffee, fruit juice,

milk and yogurt drinks. This drink is also categorised into flavoured yogurt which contains flavours and colourings. ^{4,5} Yoghurt drinks are composed of cow's milk, water, Carmine natural dyes, *Streptococcus thermophilus* and *Lactobacillus delbrueckii subsp bulgaricus* culture. ⁶ Colour changes on composite resin is more vulnerable to occur due to water or liquid absorption rate and hydrophilicity level of composite resin matrix. Excessive absorption of these substances can cause microcracks in composite resins. ⁷ Hydrolytic degradation process on the interfacial filler-matrix is caused by scission of the covalent bound. ⁸

Degradable polymer changes into oligomer and monomer, while at the same time the interfacial filler-matrix weakens and allows water to enter the interfacial filler-matrix that leads to swelling. The degradation process that happens progressively causes microstructure changes through formation of porosity, where oligomers and monomers are released. This process is known as degradation process, which causes the roughness on composite resins surfaces.9 The degradation process in the polymer matrix causes changes in surface roughness on the composite resin because it leaves filler bumps. 10 Constant consumption of acidic drinks are closely related to changes in the coarser surface of matrix polymer restoration.¹¹ The roughness of composite resin surfaces can increase bacterial growth, secondary caries, gingival inflammation and surface staining.1 The aim of this study was to determine the effect of nanofilled composite resin immersion duration using guava yogurt drink on the surface roughness and colour changes.

MATERIALS AND METHODS

This study used an experimental laboratory method with pre-test and post-test design. It used nanofilled composite resin (3M Filtek Z350XT shade A3) in cylindrical shaped with diameter Of 10 mm and thickness of 2 mm (ISO 4049 (2000)) that were made using fiberglass mould. Twelve samples were divided into three treatment groups, were soaked in yoghurt drinks for 12 hours, 24 hours, and 36 hours. The estimated length of exposure time of the composite with every 1 glass of drink consumption is 1 minute. 12 so that 12 hours of immersion is assumed to be equal with regular consumption of yogurt drinks once a day for ± 2 years, 24 hours of immersion is assumed to be equal with regular consumption of yogurt drink once a day for ±4 years, and 36 hours of immersion is assumed to be equal with regular consumption of yogurt drink once a day for ± 6 years. The making of samples and measurement of surface roughness was done at Integrated Research Laboratory Faculty of Dentistry Universitas Gadjah Mada and the measurement of colour change was done at Laboratory of Public Service Unit Faculty of Agricultural Technology Universitas Gadjah Mada.

The fiberglass walls and the base of the mold were smeared with vaseline and the composite

resin was put into the mould by using plastic instruments. A sheet of celluloid strip was placed on top of the sample and a 1 kg weight was put on top for around 5 minutes to obtain a flat and smooth surface.

Irradiation was carried out by using light curing unit for 20 seconds, then the hardened composite resin was removed from the mould. After that, the samples were put in a beaker glass containing artificial saliva with a pH of 6.8 for 24 hours at a temperature of 37 °C. Soaking in the artificial saliva was intended to adjust it to the condition of the oral cavity.¹

Samples which had been soaked in artificial saliva for 24 hours with temperature of 37 °C were divided into 3 groups randomly, then rinsed with aquadest, dried with tissues, and their surface roughness was measured using profilometer Starrett SR300 surface roughness tester. Meanwhile, the color was measured using chromameter Konica Minolta CR-400. Results of the colour measurements are the initial colour of the samples.

Guava flavoured packed in 250 mL plastic bottle in pink colour was poured into 12 conical tube with 10 ml in each tube. Specimens were put into the individual conical tubes and marked with number 1 to 4 for group A, 5 to 8 for group B and 9 to 12 for group C. Every conical tube was incubated with temperature of 37 °C. Specimens that have been treated according to the time specified were taken from the conical tube by using tweezer, rinsed using aquadest, and dried by using tissue. The next step was the surface roughness measurement and colour changes.

The differences of surface roughness between before and after treatments were calculated, then processed statistically. Data were tested using one-way analysis of variance (ANOVA) test with confidence level of 95% or (α =0.05). When the result was significant, it was proceeded with the least significance different (LSD) test.

RESULTS

The measurement of initial and final result generated the average and the standard deviation

Table 1. The average and standard deviation of immersion time in guava flavoured yoghurt drink towards the colour changes of nanofilled composite resin

| Immersion time | n | Average ± standard deviation |
|----------------|---|------------------------------|
| 12 hours | 4 | 7.47 ± 2.61 |
| 24 hours | 4 | 8.44 ± 0.85 |
| 36 hours | 4 | 12.72± 2.23 |

Table 2. The average and standard deviation of immersion time in guava flavoured yoghurt drink towards the changes in surface roughness of nanofilled composite resin

| Immersion time | n | Average ± standard deviation |
|----------------|---|------------------------------|
| 12 hours | 4 | 0.21 ± 0.20 |
| 24 hours | 4 | 0.24 ± 0.26 |
| 36 hours | 4 | 0.26 ± 0.16 |

Table 3. Result of normality test for colour changes

| Groups | Statistic | Level of freedom | Significance |
|----------|-----------|------------------|--------------|
| 12 hours | 0.981 | 4 | 0.910 |
| 24 hours | 0.903 | 4 | 0.447 |
| 36 hours | 0.894 | 4 | 0.401 |

Table 4. Result of normality test for changes in surface roughness

| Groups | Statistic | Level of freedom | Significance |
|----------|-----------|------------------|--------------|
| 12 hours | 0.797 | 4 | 0.097 |
| 24 hours | 0.767 | 4 | 0.055 |
| 36 hours | 0.814 | 4 | 0.130 |

of colour changes (ΔE) as presented in Table 1. While the average and standard deviation of changes in surface roughness nanofilled composite resin as presented in Table 2.

The colour changes of nanofilled composite resin after immersion in guava flavoured yoghurt drink showed an increasing trend from immersion time of 12 hours, 24 hours and 36 hours. The biggest changes occurred in the 36 hours of immersion time and the smallest was found in the 12 hours immersion time, while the changes in surface roughness of nanofilled composite

resin also showed an increase from immersion time of 12, 24 and 36 hours in guava flavoured yoghurt drink.

The result of normality test indicated normal distribution. Hence, since the data is scattered around the mean line, it shall be tested further using the homogeneity test. Homogeneity test was done using Levene's test with significance results of 0.088 (p>0.05) on colour changes and 0.516 (p>0.05) on changes in surfaces roughness. This result indicates that the data is homogen.

The result from one-way ANOVA test on colour changes shows an F value of 7.495 with significance level of 0.012 (p<0.05), which shows that immersion time in guava flavoured yoghurt drink has significantly affected the colour changes in nanofilled composite resin (Table 5). Meanwhile, one-way ANOVA test on changes in surface roughness shows an F value of 0,046 with significance of 0.956 (p>0.05) (Table 6). In other words, the immersion time in guava flavoured yoghurt drink brings no significant effect on the changes in surface roughness of nanofilled composite resin. LSD test result shows that there is no significant difference in the results between 12 hours group and 24 hours group. The result of the 36 hours group shows significant difference as compared to that of 12 hours group and 24 hours group.

DISCUSSION

Based on the provided data, there is an increase on colour changes and surface roughness along with the increasing immersion time. It denotes that immersion time significantly affects the colour changes of nanofilled composite resin, as indicated by the increasing surface roughness in this research. However, it is also revealed that immersion time did not significantly affect the changes in surface roughness.

Yoghurt drinks are composed of cow's milk, water, Carmine natural dyes, and *Streptococcus* thermophilus and *Lactobacillus* delbrueckii subsp bulgaricus culture.⁶ Composite resin matrix has hydrophilic properties that can influence

Table 5. One-way ANOVA test result on colour changes

| Source of variation | Number of Quadrates | Level of freedom | Mean squared | F | Significance |
|---------------------|---------------------|------------------|--------------|-------|--------------|
| Between Groups | 62.385 | 2 | 31.192 | 7.495 | 0.012* |
| In Groups | 37.458 | 9 | 4.162 | | |
| Total | 99.842 | 11 | | | |

^(*) there is a significant difference (p<0.05)

Table 6. One-way ANOVA test result on changes in surface roughness

| Source of variation | Number of Quadrates | Level of freedom | Mean squared | F | Significance |
|---------------------|---------------------|------------------|--------------|-------|--------------|
| Between Groups | 0.004 | 2 | 0.002 | 0.046 | 0.956 |
| In Groups | 0.406 | 9 | 0.045 | | |
| Total | 0.410 | 11 | | | |

Table 7. Mean difference of LSD test result on colour changes

| Immersion time | 12 hours | 24 hours | 36 hours |
|----------------|----------|----------|----------|
| 12 hours | - | | |
| 24 hours | 0.97 | - | |
| 36 hours | 5.25* | 4.27* | - |

^(*) there is a significant difference (p<0.05)

absorption and adsorption of carmine pigment and water. Nanofilled composite resin that was used in this research contains resin matrix bis-GMA, UDMA, TEGDMA, PEGDMA, and bis-EMA and the biggest hydrophilicity level composite resin matrix is TEGDMA, followed by bis-GMA, UDMA and HMDMA.¹³ Hydrophilicity level in matrix is influenced by the presence of ester group and hydroxyl group that cause water to undergo resorption inside the composite resin matrix. Next, hydrolytic degradation process happened through scission process of covalent bonds on matrix polymer till water is absorbed on the interfacial filler-matrix and caused the swelling of matrix polymer.^{8,9}

Degradation process arises progressively and may lead to microstructure changes through porosity formation until oligomers and monomers are released.⁹ The release of matrix monomers will leave filler bumps that changes the surface roughness.¹⁰

Amount of changes in surface roughness of composite resin depends on type and composition of drink, how long the drink contacts with composite

resin while consumed, and type of composite resin.¹⁴ These studies strengthen results that acid composition in yoghurt drink can cause changes in surface roughness, depending on acidity level (pH) and the immersion time of composite resin, which is above 36 hours. Hence, the colour changes and the level of surface roughness of the nanofilled composite resin may continue to increase along with the longer immersion time of more than 36 hours.

CONCLUSION

The colour changes in the nanofilled composite resin showed a significant change in the immersion time of 12, 24, and 36 hours, but the surface roughness of the composite resin had not changed significantly in that period.

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