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Research Report

Effects of glycerin application on the hardness of nanofilled composite immersed in tamarind soft drinks

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ABSTRACT

Background: *Loss of tooth structure is a consideration in the performance of restorative treatment involving nanofilled composite resins. Material polymerization factors and water absorption can affect the hardness of composite resins. Imperfect polymerization producing an oxygen inhibited layer (OIL) and causing water absorption can even compromise the hardness of nanofilled composite resins. Tamarind soft drink, on the other hand, has an acidic pH that compromises the hardness of nanofilled composite resins.* ***Purpose:*** *This study aimed to reveal the effects of glycerin application on the hardness of nanofilled composite resins immersed in tamarind soft drinks.* ***Methods:*** *The research constituted a laboratory experiment using 24 nanofilled composite resin samples with diameters of 5mm or 2mm, divided into six groups, namely: Group G, Group G AS 60, Group G AS 120, Group TG, Group TG AS 60, and Group TG AS 120. Glycerin was applied to the surfaces of three groups before curing, while the other three groups were not treated with glycerin. Finishing was subsequently conducted on all samples using a highspeed handpiece and superfine finishing bur, before they were polished with a low speed handpiece. The samples were then divided into specific groups, namely: a group with a 120-minute immersion time, a group with a 60-minute immersion time, and a group which was not immersed and maintained at a temperature of 37°C. Each sample was tested at three points using a Vickers hardness tester (VHT).* ***Results:*** *The results showed that the groups with glycerin had a higher hardness level than those groups. In addition, the non-immersed groups had a higher hardness level than those groups which were immersed. The one-way ANOVA test results confirmed that there was a statistically significant difference ($p<0.05$) between all groups.* ***Conclusion:*** *The application of glycerin to nanofilled composite resins immersed in tamarind soft drinks can increase their hardness levels.*

Keywords: *glycerin; hardness of nanofilled composite resin; tamarind soft drink*

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INTRODUCTION

Loss of tooth structure due to erosion, enamel abrasion and dental caries is one factor leading to restorative treatment.[1] One restoration material frequently used to replace the function of missing tooth structures is composite resin which offers the advantages of promoting attractive aesthetics of the anterior teeth and the greater abrasive resistance of the posterior teeth.[2,3]

Composite resin comprises three main components, namely: matrix resin, filler and silane coupling agent. Matrix resin consists of bisphenol A-glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA) and tryehtyleneglycol dimethacrylate (TEGDMA). Composite resin filler, ranging from traditional composite resins (macrofillers), microfillers, flowables, packables, hybrids to nanofillers, particularly with regard to its particle size has been improved. Nanofillers are composed of smooth particles with the result that the restoration possesses a smooth surface and is aesthetically attractive.[4] However, nanofillers suffer from certain disadvantages, one of which is their higher hydrophilic properties compared to other larger types affecting the water absorption of composite resin.[5]

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Composite resin can experience changes in its mechanical properties due to oral conditions, such as salivary pH and food intake.[6] One such vatable property is hardness[7] which can, consequently, be considered a measure of the resistance to wear of restoration materials since it can influence mechanical friction during mastication and tooth brushing.[8] The properties of composite resin potentially affecting its hardness include: water absorption, composite resin hardness, irradiation distance and material polymerization.[8]

The polymerization of composite resin materials can be disrupted when their surfaces are exposed to air. The disturbance causes obstruction of the polymerization process, producing an oxygen inhibition layer (OIL) which can then affect the prognosis of composite restorations because it reduces surface hardness, durability and marginal adaptation. To minimize the occurrence of OIL, glycerin inhibitors can be employed during the curing process. Glycerin is stable in a medium of atmospheric oxygen because, when exposed to air, the glycerin will be in equilibrium with the water vapor (relative humidity) in the surrounding atmosphere. Therefore, glycerin bonds and the surrounding objects will not experience changes at normal temperatures. Hence, glycerin can be used as a barrier to prevent the formation of OIL on occlusal surfaces or those of composite resins that are difficult to access.[9]

Another factor affecting the hardness of composite resins is the absorption of water present in the food and beverage consumed daily by patients and in direct contact with tooth surfaces.[10] Soft drinks constitute one popular beverage consumed by the Indonesian public with annual per capita consumption within the country amounting to 33 liters.[11] Such drinks are non-alcoholic processed liquids containing food ingredients or other additives, both natural and synthetic, which are packaged ready for consumption.[12]

Java tamarind, on the other hand, contains several kinds of acid, including citric acid, in addition to antioxidant compounds which donate H atoms from their phenolic groups, thereby promoting increased antioxidant activity. Since they contain a variety of healthy ingredients, the consumption of tamarind-derived soft drinks has increased in popularity.[13]

Such beverages are considered to be soft drinks with a pH of 3.7.[12] This level of acidity causes greater micromorphological damage to the composite resin.[10] Consumption of acidic drinks can also dissolve the composite resin since it contains numerous H ions capable of continuously eroding the composite resin material. This subsequently leads to the degradation of the composite resin component.[7] Moreover, it also affects its users by compromising surface hardness[8] through the process of polymerization triggered by glycerin and water absorption of composite resins. This study aimed to reveal the effects of glycerin application on the hardness of nanofilled composite resins immersed in tamarind soft drinks.

MATERIALS AND METHODS

This research constituted an experimental laboratory study featuring a post-test only control group design. There were six groups, namely: group G to which glycerin had been applied; group G AS 60 to which glycerin had been applied and which was immersed for 60 minutes; group G AS 120 to which glycerin had been applied and which was immersed for 120 minutes; group TG which was glycerin-free; group TG AS 60 to which no glycerin had been applied and which was immersed for 60 minutes; and group TG AS 120 which was glycerin-free and which was immersed for 120 minutes. Each of the groups consisted of four samples. The immersion time was determined by calculating the duration of the contact between nanofilled composite resins and drinks consumed in each package/day (one minute) for 30 days. The estimated total amount of time required when consuming drinks is one month and two months, which in this study were converted to 30 minutes and 60 minutes.[7] The number of samples for each group was based on Federer's formula: $(n-1)(t-1) \geq 15$. Consequently, with t (number of groups) being 6, the number of samples in each treatment group was four.

Nanofilled composite resin (FiltexTM 3M ESPE Z 350 XT) was produced with a plastic 5 mm x 2 mm sized ring mold that had been inserted into a brass disc and subsequently applied using plastic filling instruments and condensed using a cement stopper. 0.5 ml of glycerin was applied with a microbrush during each treatment before curing (F LED-B, Woodpecker, USA) was performed for 20 seconds at a distance of 0 mm, forming a plane perpendicular to the resin surface. Finishing was carried out using a highspeed handpiece (S MAX M, NSK, USA) and superfine finishing bur (314 C 850, Edenta, Switzerland). The samples were then polished using a polishing kit (CW 351 4, Ra 0309 Tobuom, China).with a low speed handpiece (Type EX, NSK, USA) at 15,000 rpm for one minute in the same direction to obtain the same pressure on each side.

Immersion in 20 ml of tamarind soft drink (Ultra Jaya Tbk, Indonesia) contained in glass beakers was conducted, all samples being inserted using tweezers with the entire upper surface of each sample being immersed. The glass beakers were then covered with aluminum foil. Following immersion 60 or 120 minutes in duration or no immersion, the samples were incubated at a temperature of 37ºC.[7]

The hardness of each sample was subsequently measured at three points using a Vickers hardness tester (VHT, 402MVD, Wilson®, USA). The points were determined from the center of the composite resin before being shifted 1mm to the right and left and indented on the section focused on the lens. Indenting was performed at a pressure of 100 gf for 15 seconds. Hardness testing on each sample was conducted on the upper surface of each sample. The pressure exerted by the indenter centered on the three points positioned in line and focused on the observation lens. The hardness results were determined

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after the application of vertical and horizontal pressure through the observation lens and calculated as an average per group.[14]

The normality of the data was analyzed using a Shapiro-Wilk test, while a Levene's test assessed its homogeneity producing a significance level of p>0.05. Since the data was normally and homogeneously distributed, it was analyzed using a one-way ANOVA test. An LSD test with a significance level of p>0.05 was subsequently conducted to observe differences between the groups.

RESULTS

The results of the research showed that the average Vickers hardness number (VHN) across all groups with glycerin was higher than that in those without glycerin. Moreover, in all groups with time variations it decreased as the duration of immersion became more extended (Table 1). This research indicated differences in the average VHN between all treatment groups (Table 2). The highest VHN average occurred in the group with glycerin (G), while the lowest average was recorded by the group without glycerin (TG AS 120) with an immersion time of 120 minutes.

The results indicated differences in the average VHN between the treatment groups featuring glycerin application and those without. The groups with glycerin application (G, G AS 60 and G AS 120) had a higher VHN than those without (T, TG AS 60 and TG AS 120). There were also differences in the average VHN between the groups with glycerin application and those groups without after the same immersion time (Table 3).

The results of this research revealed differences in the average VHC between the treatment groups with differing immersion times (without immersion, 60-minute immersion, and 120-minute immersion). The group with a 120-minute immersion time recorded the lowest average VHN with the result that it recorded the highest difference in the average VHN compared to the group without immersion. Moreover, the difference in the average VHN of the 60-minute immersion group and that of the 120-minute immersion group indicated that there was a decrease in the average VHN based on the duration of immersion.

The data obtained was analyzed by means of a Shapiro-Wilk normality test the results of which showed a significance value greater than 0.05, thereby indicating that the data was normally distributed. The results of a subsequent Levene's test indicated a significance value of 0.058 (p>0.05) and confirmed the homogeneity of the data.

Table 4 shows that a one-way ANOVA parametric test produced a significance result of 0.000 (p<0.05) indicating differences between the test groups. Subsequent multiple post-hoc comparison tests followed by an LSD test produced a significance result of 0.000 (p<0.05) across all groups with the exception of groups G AS 120 and TG AS 60 (0.002). Therefore, significant differences can be said to have existed between treatment groups.

Table 1. The average of VHN in all groups

Groups	The average of VHN
G	98.12 ±0.46
G AS 60	67.34 ±0.85
G AS 120	61.10 ±1.39
TG	72.24 ±0.91
TG AS 60	63.72 ±1.21
TG AS 120	54.38 ±0.9

Table 2. Differences in the average of VHN based on glycerin application

Treatment groups		Difference in the average of VHN
G	TG	25.88
G AS 60	TG AS 60	3.62
G AS 120	TG AS 120	6.72

Table 3. Differences in the average of VHN based on immersion

Treatment groups		Difference in the average of VHN	Differences in the average of VHN between immersion times
Glycerin	Immersion for 60 minutes	30.78	6.24
	Immersion for 120 minutes	37.02	
Without glycerin	Immersion for 60 minutes	8.52	9.4
	Immersion for 120 minutes	17.92	

Table 4. One-way ANOVA parametric test results

	Sum of Squares	df
Between groups	4648.555	5
Within groups	18.098	18
Total	4666.653	23

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DISCUSSION

The results showed that the groups using composite resins without glycerin application, but with varied immersion times, had lower VHN than the groups using composite resin with glycerin application and varied immersion times. The average VHN values in the group with a 120-minute immersion time was lower than in the 60-minute immersion group and in the non-immersion groups due to the nature of the effect of the polymerization process on surface hardness and water absorption of composite resins.

Table 2 indicates that the reduction in the surface hardness of the glycerin-free groups was influenced by several factors including the degree of polymerization conversion and the presence of OIL layers. The degree of conversion resulting from the polymerization process is the percentage of carbon metal methacrylate double bonds capable of binding to free radicals and producing a single bond which forms a polymer chain. Up to 50-70% of C = C covalent bonds can be converted to produce approximately 30-50% of metal methacrylate which do not undergo initiation with free radicals.[2] This is also supported by the glycerin-free treatment with the result that free radicals bind with oxygen to produce peroxyl radicals:[15]

$$R \bullet + O2 = R\text{-}\ OO \bullet$$

Free radicals + Oxygen = Peroxyl radicals

Free radicals that bind to oxygen reduce the number of free radicals that should bind to the covalent C = C bond in metal methacrylate.[2] The results of monomers that are not bound to free radicals forming OIL when combined with the remainder of the unconverted methacrylate monomer can induce changes in the surface hardness of the composite resin.[15]

During this research, a droplet of glycerin was applied to the surface of the composite resins. Its consistency did not harden during curing and had a transparent color which did not affect the irradiation distance and intensity of the LED light during polymerization. The application of glycerin is intended to prevent the bond between free radicals and oxygen in order to increase the surface hardness of composite resins.[15]

Other factors affecting polymerization of composites include: exposure time, exposure distance and composite resin thickness.[8] All groups were irradiated for 20 seconds as recommended with a irradiation distance of 0 mm in the perpendicular position and with the same thickness of 2 mm. These factors were used as dependent variables applied to all samples in order that they did not affect the hardness of the composite resin.

The hardness of nanofilled composite resins is also influenced by water absorption.[8] The specific resin used in this research was Filtex TM Z350 XT transluscent shade which contains bis-GMA, UDMA, TEGDMA and bis-EMA resin. TEGDMA constitutes one of the comonomers possessing the largest hydrophilic properties at 69.51 µg/mm^3. UDMA monomers also contain the element O which is electronegative with the result that it tends to attract OH groups which release H+ from water.[16] This research employed a combination of non-agglomerated and non-aggregated fillers which included silica 20 nm in size and zirconia 4-11 nm size. Non-aggregated fillers are ones that do not undergo central collection in a specific area with the result that they possess large surfaces.[2] The surface of zirconia filler is porous which facilitates the absorption of water by the composite resins.[17]

Water absorption in composite resin subsequently causes degradation of composite resins which involves the loss of chemical structure in composites such as Bis-GMA. This is due to various factors including hydrolysis and water-related environmental influences.[6] In the composite resin groups involving immersion in tamarind drinks, the surface hardness was lower than that in the groups without immersion in tamarind drinks. This indicates that food and beverages consumed can affect the hardness of composite resins.

Tamarind soft drinks have an acidic pH of approximately 3.7. Low pH drinks (3-6) damage the resin surface. Hence, the pH value can be an indicator determining H+ ion content since at low pH levels it will be higher.[5] H+ ions are absorbed into the matrix and react with the ester group of dimethacrylate monomers, forming carboxylic acids and alcohol molecules. Dimacrylate monomers that bind to H+ ions break down the double bond of the resin monomer into a single bond and produce OH-. This causes expansion of the material, softening of the matrix and enlargement processes.[17,18]

In addition, water absorption caused by immersion also results in degradation of siloxane bonds (bonds between silanol groups on the surface of silica and silane coupling agents) through hydrolysis reaction. Water in contact with the surface of silica particles can break the bond of siloxane and subsequently trigger a bond between the particles of the filler material which can increase weight loss in the composite resin. Consequently, this affects the bond between the filler and the resin matrix causing it to become unstable.[19] Bonds are released and the material becomes porous. The release of this filler then causes numerous small gaps in the composite resin with the result that it becomes less dense, thereby reducing its mechanical properties, namely its hardness.

One of the acidic elements within the composition of soft drinks derived from tamarind is citric acid containing electropositive H+ ions which are readily attracted to the double bond O with electropositive properties. This condition can promote degradation of the bis-GMA monomer and siloxane bonds by a similar mechanism to that produced by hydrolysis. This process can subsequently cause reduced hardness in composite resins.

It can be argued that the longer the immersion time, the lower the average VHN. The consumption of acidic drinks, such as soft drinks and fruit juices, can actually reduce the hardness of the composite resin due to surface

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damage caused by material they contain.[20] Immersion of composite resin for 60 minutes and 120 minutes resulted in a gradual decrease in hardness depending on the length of the immersion time. The decrease in the average VHN was due to water absorption requiring a significant period to reach equilibrium. Consequently, the lowest hardness value was registered by the group with an immersion time of 120 minutes, but without the application of glycerin.[9,14] It can be concluded that the application of glycerin is capable of increasing the hardness of composite resins immersed in tamarind soft drinks.

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