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# An evaluation of the effect of different solutions on the microhardness of aesthetic restoration

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## Abstract

Dietary awareness is an important issue in modern society. The consumption of carbonated drinks is popular with the youth of today and the habit is carried over into adulthood. One of the most important properties that determine the durability of dental materials in the oral cavity is their resistance to dissolution or disintegration and the longevity of dental restorations depends on the durability of the material and its properties, such as wear resistance, durability of the interface between tooth and restoration and the level of tooth destruction.

The aim of this study was to measure surface hardness of composite after storage in dry condition and after immersion in saliva and solutions that represent the popular diet.

Fifty disk shaped specimens (5×2mm) were prepared from Z100 universal composite using polytetraflouroethylen mold. The specimens were divided into 5 groups (n=10) and stored for 7 days at 37 ̊C in different types of storage conditions (dry condition, artificial saliva, coca cola, heptane and citric acid). After storage, Brinell Hardness Numbers were calculated. The data were analyzed with one – way analysis of variance (ANOVA) and LSD test.

There was a highly significant difference when comparing the storage agents. Coca cola and citric acid solutions produced the highest reduction in microhardness of composite. It was followed by heptanes, artificial saliva. The storage of specimens in dry container produced the highest Brinell Hardness Numbers.

Microhardness of composites stored in dry condition was the highest value, followed by artificial saliva, heptane solution, cola soft drink and citric acid . Acid solutions have the potential to erode composite resin material and this reduction was highly significant, which means that people who consume acidic foods and drinks excessively should be aware of their esthetic restorations.

**Key words: Brinell hardness number, food simulating solution, storage solutions.**

## Introduction

Resin-based composites are becoming more popular in restorative dentistry, particularly because of their superior esthetic outcomes. They typically consist of a methacrylate-based resin matrix (mass fraction of about 25–30%), glass or ceramic fillers (mass fraction of about 70–75%), and a filler-matrix coupling agent[1] .The clinical success of an aesthetic direct restorative material depends upon its mechanical behavior, physical and chemical characteristics, as well as clinical indications.[2] One of the most important properties that determine the durability of dental materials in the

oral cavity is their resistance to dissolution or disintegration. [3] Acid erosion has a clinical significance because acidic conditions can occur orally either due to the ingestion of acidic foods or the degradation of poly saccharides to acids in stagnant areas of the mouth.[3] Dietary awareness is an important issue in modern society. The consumption of carbonated drinks is popular with the youth of today and the habit is carried into adulthood. Healthy diets such as fruits, fruit juices and yogurt may as well cause erosion by their acidity.[4] The consumption of acidic foodstuff and beverages plays a major role in the development of erosion. Along with the change of lifestyle through the decades, the total amount and frequency of consumption of acidic foods and drinks have also changed. The potential erosive effect of a soft drink depends on a number of conditions such as pH and buffering capacity, type of acid, adhesion of the product to the dental surface, chelating properties and calcium, phosphate and fluoride concentration.[5]

In the oral environment, it can be assumed that saliva, food components, beverage and interaction among these materials can degrade and age dental restorations. The resin matrices of dental composites become softened with exposure to organic acids and to various food and liquid constituents. In addition, when composites are soaked in oral fluids, the disintegration at the resin – filler interface occurs. Therefore, the chemical environment in the oral cavity may have a critical influence on the in vivo degradation of composite resins. [6] The longevity of dental restoration depends on durability of the material and its properties such as hardness and wear resistance.[5] Hardness is defined as the resistance of a material to indentation.[7] Surface hardness correlates well to compressive strength, and abrasion resistance.[8]

Conventional composite were found to have significantly lower wear resistance once they were immersed in chemicals which softened the resin matrix, copolymer. The physical properties of composites are, however, not just dependent upon the nature of resin matrix, but also upon the inorganic reinforcing filler and the resin – filler interface. Under oral conditions, the silane coating at the resin – filler may also disintegrate.[9]

Oral cavity is a complex environment where the material is in contact with saliva, a fluid that contains a variety of inorganic and organic species, together with bacterial flora complex. [8]

Alcoholic and/or acidic environment cause the surface degradation of resin composites. In addition, the surface degradation of resin materials is related to the content of fillers, distribution of the fillers, and composition of the matrix resin and the effect of silane surface treatment on the fillers.[3]

The longevity of dental restoration depends on durability of the material and its properties such as hardness and wear resistance. [5] Surface hardness correlates well to compressive strength, and abrasion resistance [8] and hardness is defined as the resistance of a material to indentation. [7]

The objective of this in vitro study was to measure surface hardness of composite after storage in dry condition and after immersion in artificial saliva and solutions that represent popular diet.

## Materials and Method

A commercially available light cured restorative material Z100 shade A2 was used. The chemical

composition of the material is listed in Table 1. [10]

A poly tetrafloroethylen mold is used to prepare fifty disc shaped specimen having a dimensions of 5mm in diameter and 2 mm in depth. The material is placed in the mold, covered with a Mylar strip and then light cured. Light curing is performed with quartz – tungsten - halogen lamp (Astralis 3; Ivoclar Vivadent Schaan, Liechtenstien), having a power density of 600mW/cm2 and the light tip was in close contact with the Mylar strip during polymerization. Curing was done for 40 seconds according to the manufacturer's instruction. All of the specimens were randomly assigned into four test group and one control (n=10). The control group (D) was stored in dry container in the incubator at 37Cº for seven days (dry condition).

The test groups were conditioned into one of the four media and stored at 37Cº for seven days in the incubator: 1. artificial saliva (AS), 2. Coca cola (C), 3. Heptane (H), 4. Citric acid (CA) and the pH values of the test drinks were checked using a pH test paper as in Table 2.

The chemical composition of artificial saliva was chosen according to Al – Samaraai [11] and Luthy et al.,[12] Table 2.

At the end of the conditioning period, each specimen was removed from the container, rinsed with running water for 30s and then air dried. Then Brinell hardness test was done using a load of 100gm with a dwell time of 10 seconds. The microhardness was calculated in $kg/mm^{2.}$ And it was calculated according to the following equation [7]:

$$BHN = 0.102 * L / \pi D/2(D\sqrt{D^2-d^2})$$

$\pi = 22/7$

D =2.5

d = Diameter of Indentation for each specimen

L = Load applied by Newton

1 Newton = 0.102 kilogram

One way analysis of variance (ANOVA) test was done to evaluate the effect of different solutions on surface hardness of Z100 universal composite restorative material. If there is significance among groups, then it followed by least significance difference (LSD) test.

## Results

Mean and standard deviation of Brinell hardness number are presented in Table 3. Brinell hardness number for group D was the highest, Followed by group AS, group H, group C, while group CA produced the lowest value.

One – way ANOVA test in Table 4 revealed that there was a highly significant difference when comparing among storage media and then LSD test was performed. From LSD test in Table 5, it can be seen that there is a highly significant difference when comparing among groups, except when comparing between group C and CA, there was no significant difference between them. There was a significant difference between group AS and group H.

## Discussion

Under oral conditions, composite resins may be exposed either intermittently or continuously to chemical agents found in saliva, food and beverages. Intermittent exposure occurs during eating or drinking until the teeth are cleaned. On the other hand, continuous exposure may occur when the chemical agents are absorbed by adherent debris (such as calculus or food particles) at the margins of restorations or produced by the bacterial decomposition of debris.[13] Besides, these chemical agents can be trapped around the margins of

inadequately finished restorations or restorations finished with an overflow of dental materials. In addition, food particles at the margins of restorations may serve as reservoirs for these chemicals.[6] The chemical environment is one aspect of the oral environment, which could have an appreciable influence on the in vivo degradation of composite restoratives.[13] Many foods and drinks (e.g. water, acids, soft drinks, food derivatives) affect the behavior of restorative materials[3] since chemical softening of restoratives may result in decreased physico-mechanical properties.[14]

Hardness is defined as the resistance to permanent indentation or penetration. It's used to predict the wear resistance of a material and its ability to abrade opposing dental structure. Among the properties that are related to the hardness of a material are strength, proportional limit and ductility.[15]

In this study, four conditioning media were used and their effect on microhardness of Z100 universal composite was measured. These conditioning media were: artificial saliva, heptanes, citric acid and cola. The conditioning of specimens in dry container was used as a control. These food simulating liquids used for conditioning the restorative material were chosen according to FDA guidelines.[16] Heptane simulates butter, fatty meats and vegetable oils. The citric acid conditioning simulates beverages, including vegetables, fruits, candy and syrup.

As the greatest change in hardness had been shown to occur within the first seven days of exposure to solutions and the hardness of composite is affected by conditioning them in solutions for seven days[13, 6, 1], this period of storage was selected for the present study.

The results of the present study revealed that the specimens stored in dry container had the highest surface hardness measurements, this agrees with Medeiros et al., [17], who found that mechanical properties for specimens stored in dry container were superior than other groups and agrees with Yap et al., [18] who found the highest hardness values for specimens stored in dry container for seven days.

This result is also in agreement with Tsuruta and Viohl in [19], who found that surface hardness increased with time when specimens were stored in dry container.

For all specimens stored in the four storage media, there was a significant reduction in microhardness of the Z100 composite and such reduction was not the same. This reduction in microhardness after conditioning in solutions is because these modern tooth colored restoratives have been shown to behave differently in solutions [20] and many foods and drinks (e.g. water, acids soft drink, and food derivates) affect the behavior of restorative materials.[4]

In composites, polymerization shrinkage and diffusion of moisture through the resin component lead to the initiation and propagation of microcracks in the resin matrix. This process could provide a supply of chemical agents and a path for further diffusion into the restorative material, thereby resulting in more rapid degradation.[1]

Water or other liquids enter the polymer network through porosities and intermolecular spaces. The uptake of water or other solvents by dental composites may cause expansion that can affect the dimensions of the restorations. The solvent diffuses into the network and separates the polymer chains, creating an expansion. In addition, water uptake is accompanied by a loss of unreacted components, like

unreacted monomers, or ions from filler particles.[21] The reduction in hardness is a consequence of the separation of the polymer chains by a molecule that does not form primary chemical bonds with the chain, but simply serves as a space occupier (i.e. plastification). Thus the main effect of the solvent is to reduce interchain interactions, such as entanglements and secondary bonding.[17]

Another possibility could be the chemical degradation occurring via hydrolysis. After water or solvents enter the polymer bulk, the intrusion of water triggers chemical polymer degradation, leading to the creation of oligomers and monomers. [22]

Such progressive degradation changes the microstructure of the composite bulk through the formation of pores, via which oligomers, residual monomers, degradation products and additives are released.[17]

In this study, when comparing microhardness of Z100 composite resin among storage media, the storage in artificial saliva produced the highest surface hardness. This finding agree with Martins de Oliveira et al., [23] who explained this result by the deposition of minerals on the surface of specimens, resulting in the formation of film probably composed of calcium.

This result agrees with Aliping – Mckenzie et al., [24], who measured microhardness of composites after conditioning in artificial saliva, coca cola, apple juice and orange juice.

Storing specimens in heptane led to a significant reduction in microhardenss of composite. This reduction is more than specimens stored in dry container and artificial saliva, but it is less than the reduction for specimens stored in citric acid and phosphoric acid.

This reduction is because the resin matrix can be potentially damaged by organic solutions (heptane and aqueous ethanol solution).[6,1]

The inorganic fillers, on the other hand, can be damaged by water and citric acid.[1] In this study, Z100 composite resin was used which was composed of 85% by weight inorganic filler. This could explain why the reduction in microhardness was greater in both acids solutions than heptane solution.

The results of the present study disagree with Yesilyurt et al., [1] who claimed that there is a slight increase in hardness noted for all composite resin specimens conditioned in heptane, although this increase was not statistically significant. They claimed that heptane reduced oxygen inhibition during post-curing and eliminated leaching of silica and combined metal in fillers, which occurred from conditioning in aqueous solutions. They suggested that further studies would be needed to be conducted in order to have a more thorough understanding toward the increase in microhardness after conditioning in heptane solution.

The results of the present study disagree with Akova et al., [6], who measured surface hardness of provisional restorations in water, citric acid, heptane and ethanol solution. The hardness of all tested provisional restorations showed a reduction in heptane solution. The difference in this result and the result of the present study is due to the difference of chemical composition of the tested materials.

In this study, when comparing between specimens stored in cola and those stored in citric acid, there was no significant difference between them. This is because acids adversely affect the surface integrity of resin based restoratives; since cola contains phosphoric acid and both acids (citric and phosphoric acids) are erosive.[20]

The reduction in microhardness after conditioning in acids could be explained that fillers tend to fall out from resin material[25] and the matrix components decomposes when exposed to low pH environment.[26] Many soft drinks are acidic and the pH is 3 or lower. This means that drinking acidic drinks over a long period and with continuous sipping can erode the tooth enamel and the resin material as well.[3] In this study, the pH value for citric acid was 2.5, and for cola solution was 2.4. The resin based composites were found to undergo greater micromorphological damage following an acid challenge and these acids adversely affect the surface integrity of resin composite. In the oral environment, the effect of other solvents and esterase may have a more detrimental and sustained effect on the mechanical properties of dental composites. The deleterious effect of weak intra oral acids on inorganic filler may also contribute to decreased mechanical properties.[6,1]

Another explanation for the reduction in microhardness of Z100 composite resin after condition in both acids (citric and phosphoric) is the composition of Z100 composite. The resin of it is BisGMA and TEGDMA. According to Rios et al., [5], the acid could attack the resin due to the softening of bisphenol-A- glycidyl methacrylate (Bis – GMA) polymers, which could be caused by leaching of the diluents agents such as triethylene glycol dimethacrylate (TEGDMA). The resin matrices of dental composites are softened by organic acids and various foods and liquid constituents. Leaching of composite fillers and disintegration of the filler – resin interface (silane coupling agent) can also occur under oral conditions.[18]

In addition, degradation of the inorganic filler may also play a role in the reduction of microhardness. The leakage of filler constituents has been shown to produce cracks at the resin–filler interface, which may lead to weakening of the material.[6] Acids have a greater potential to produce expansion of Bis – GMA based polymers and the acid solutions showed a tendency to negatively affect the mechanical properties of the composite. The degradation products of polymers generally show that extraction of monomers and oligomers is more complete in alcohol or organic solvents compared to water.[17]

This result agree with Lim et al., [27] and Han et al.,[3], who found that any portion of the organic matrix resin which is insufficiently polymerized can be dissolved by alcoholic and acidic solutions, and particles can thus easily fall out. They gave another possible cause of surface degradation which was that the filler and matrix resin were too weakly bonded. This might be related to the surface treatment of fillers, whereby insufficient surface treatment with silane was thought to result in filler erosion.

The result of this study agrees with Wongkhantee et al., [4], who stated that food stuffs with lower pH have greater erosive effect. And tooth-coloured filling materials reportedly display a tendency to erode under acidic conditions. They stated that organic acids were found to induce softening of bis-GMA based polymers.

However, these results disagree with Yanikoglu et al., [20], who measured microhardness of composite after conditioning in phosphoric and citric acid. They claimed that although both acids are erosive, but citric acid is more aggressive than phosphoric acid and they said it is not clear why phosphoric acid is less aggressive.

The results of the present study disagree with Yesilyurt et al. [1], who claimed that hardness and flexural strength of all tested composites were

not significantly changed after conditioning for 7 days in citric acid. They suggested that further studies are needed to investigate and elucidate the effects of citric acid conditioning on the hardness of composite resin.

The results of the present study disagree with Yap et al.[13], who measured surface hardness of several types composite resin in artificial saliva, citric acid, lactic acid, ethanol and heptanes. They suggested that there was an increase in hardness of composite after storage in artificial saliva and citric acid, but there was a reduction in the hardness following storage in heptanes solution. The difference in the results of Yap et al., [13] and the results of the present study is the methodology of specimens' preparation. In the present study, following specimens' polymerization, they were immediately conditioned in the conditioning solution and in the dry container without any treatment. In Yap et al [13] methodology, the specimens were stored in artificial saliva for 24h following polymerization to allow elution of unreacted components from the composite and to allow for post cure. Then the specimens were conditioned in the storage media for 7 days. In the present study, elution of leachable unreacted components was in the storage media so which leads to differences in the results between this study and Yap et al.,[13].

## Conclusion

The microhardness of universal composite Z100 was evaluated after seven days storage in dry conditions and different food simulating solutions. Within the limitation of this in vitro study, the following conclusions were drawn:

1. Microhardness of composites stored in dry condition was the highest value, followed by artificial saliva, heptane solution, cola soft drink and citric acid.
2. Acid solutions have the potential to erode composite resin material and this reduction was highly significant, which means that people who consume acidic foods and drinks excessively should be aware of their esthetic restorations.

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Table 1: Materials used in this study

| Material | Manufacturer | Type | Resin | Filler | Filler size | Filler content (by volume) | Filler content (by weight ) |
|---|---|---|---|---|---|---|---|
| Z 100 | 3M Dental Products, St.Paul, MN, USA | Minifilled | BisGMA TEGDMA | Zirconia silica | 0.5 – 0.7 (mean) | 66% | 85% |

Table 2: Solutions used in this study

| Solution | Manufacturer | pH |
|---|---|---|
| Coca cola | C C Beverage limited, Efraz Road, Kawrgosik-GradaRash, Erbil Iraq. | 2.4 |
| Heptane | Al-Ansari for antiseptics, Aleppo, Syria | 5 |
| Citric acid | Alzaidan Scientific Bureau. | 2.5 |
| Artificial saliva | NaCl 0.4g/L, KCl 0.4g /L, $CaCl_2.2H_2O$ 0.795g/L, $NaH_2PO_4$ 0.69 gm/L, Urea 1.0 g/L, Distilled water 1000 ml | 7 |

Table 3: Mean and standard deviation SD of all groups

| Group | Mean and SD |
|---|---|
| Group D | 67.767 ± 2.104 |
| Group AS | 40.682 ± 1.566 |
| Group C | 30.895 ± 0.284 |
| Group H | 38.715 ± 0.784 |
| Group CA | 30.600 ± 0.479 |

Table 4: One – Way ANOVA test among the data of Brinell Hardness Number for each group

| Group | F - Value | P – Value | Significance |
|---|---|---|---|
| Group D | 1488.517 | P<0.001 | HS |
| Group AS | | | |
| Group C | | | |
| Group H | | | |
| Group CA | | | |

HS: Highly significance at P <0.001

Table 5: LSD test comparing among groups

| **Group** | **Difference between means** | **P – Value** |
|---|---|---|
| D &AS | 27.085 | P<0.001 |
| D & C | 36.872 | P<0.001 |
| D & H | 29.052 | P<0.001 |
| D&CA | 37.166 | P<0.001 |
| AS & C | 9.787 | P<0.001 |
| AS & H | 1.967 | P<0.01 |
| AS & CA | 10.081 | P<0.001 |
| C & H | -7.819 | P<0.001 |
| C & CA | 0.2948 | P>0.05 |
| H & CA | 8.114 | P<0.001 |

P<0.001 HS
P<0.01 S
P>0.05 NS