An *in vitro* evaluation of the effects of different acidic beverages on the surface hardness of restorative materials

Farklı asidik içeceklerin restoratif materyallerin yüzey sertliklerine olan etkilerinin *in-vitro* olarak değerlendirilmesi

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SUMMARY

Aim: The aim of this study was to examine the effects of different acidic beverages on the surface hardness of restorative materials which are frequently used in clinical practices.

Materials and Methods: In this study, cola, sour cherry juice, apple juice, energy drink and orange juice were used as acidic beverages while two composite resins (Filtek Silorane, Filtek Z-550), one flowable composite (Filtek Ultimate Flowable), one compomer (Dyract Extra) and one resin modified glass ionomer (Fuji II LC) served as restorative materials to be tested. After measuring the initial surface hardness of the samples with Barcol surface hardness tester, each sample was put into acidic beverage for five seconds after which they were placed in artificial saliva for five seconds. This cycle was repeated ten times daily for one month. The control group was stored in the artificial saliva without any exposure to this cycle. At the end of the one-month-period, surface hardness of the restorative materials were measured again.

Statistical analysis used: "Repeated Measures Analysis of Variance" and "Tukey's Multiple Range Test" were used for statistical analysis.

Results: The restorative materials were affected by the acidic beverages. The liquids used in the study, except the artificial saliva, had statistically similar effects on the surface hardness values of the materials used ($p<0.05$).

Conclusion: In conclusion, the beverages used in this study affected the surface hardness of the restorative materials investigated differently.

Keywords: Surface Hardness, Acidic Beverages, Resin Composite, Compomer, Silorane

ÖZET

Amaç: Bu çalışmanın amacı; farklı asidik içeceklerin kliniklerimizde sıkça kullanılan restoratif materyallerin yüzey sertlikleri üzerine etkilerini incelemektir.

Gereç ve Yöntem: Bu çalışmada asidik içecekler olarak kola, vişne suyu, elma suyu, enerji içeceği ve portakal suyu; restoratif materyal olarak ise iki adet kompozit rezin (Filtek Silorane, Filtek Z-550), bir adet akışkan kompozit (Filtek Ultimate Flowable), bir adet kompomer (Dyract Extra) ve bir adet rezin modifiye cam iyonomer (Fuji II LC) kullanıldı. Başlangıç yüzey sertlik değerleri Barcol yüzey sertlik ölçüm metodu ile ölçüldükten sonra her bir örnek 1 ay boyunca toplam 10 döngü olmak üzere; 5 sn asidik içecek, 5 sn yapay tükürükte bekletildi. Kontrol grubu, örnekler döngüye tabi tutulmaksızın yapay tükürük içerisinde saklandı. 1 ayın sonunda restoratif materyallerin yüzey sertlikleri tekrar ölçüldü. İstatistiksel değerlendirme "Tekrarlanan Ölçümlü Varyans Analizi" ve "Tukey çoklu karşılaştırma testi" ile yapıldı.

Bulgular: Restoratif materyaller asidik içecekten etkilenmiştir. Deneyde kullanılan sıvılardan yapay tükürük dışındaki sıvılar, kullanılan materyallerin yüzey sertlik değerleri üzerinde

istatistiksel olarak benzer etki göstermiştir (p<0,05).
Sonuç: Sonuç olarak, bu çalışmada kullanılan içecekler, incelenen restoratif materyallerin yüzey sertliklerini farklı şekillerde etkilemişlerdir.
Anahtar Kelimeler: Yüzey sertliği, asidik içecekler, kompozit rezin, kompomer, Silorane

INTRODUCTION

One of the most significant contributions to dental technology has been the introduction of adhesive dentistry.[1] The composition of the resin-based composites have changed significantly since their introduction to the market. One of the most significant of these changes is the reduction of the filler size and the increase in the amount of it, which, in turn, enabled the production of materials with higher wear resistance and easier polishability. [2,3]
Nanohybrid composites were produced by adding particles of nano size to microhybrid composite resins[4] In addition to the ad¬vantages of nano fill and/or nanohybrid resin composites such as strength, low wear, and polishability, these mate¬rials generally have different shades, allowing the natural dental tissue reproduction in an efficient way with the stratification technique.[1]
One of the disadvantages of composite resins is polymerization shrinkage[5] “Silorane” monomer, whose name was derived from its chemical components, namely, siloxane and oxirane, was developed to overcome this drawback. [4,6,7] Silorane based composite resin, which is argued to be more resistant to saliva, water and chemicals in the oral cavity, was developed particularly for the restoration of posterior teeth.[8]
Flowable composites are generally produced with low viscosity. Since their filler content is low, their mechanical properties are slightly reduced.[3,7]
Polyacid modified composite resin, commonly known as compomer, was first developed in the early 1990s. It was introduced as a new class of restorative materials which combined the aesthetics of conventional composite resins and, the fluoride release and adhesion of glass ionomer cements. [9]
Resin modified glass ionomers (RMCI) are hybrids of glass ionomers and composite resins. It was developed as a result of efforts to improve the mechanical properties of glass ionomer cement (GIC). These efforts also aimed to reduce the curing time and vulnerability of GIC to moisture.[10-12]
One of the most important physical properties of restorative materials contributing to their clinical success is surface hardness, which also sets the mechanical properties of restorative materials.[13,14] Barcol hardness test is based on resistance to indentation. Barcol hardness tester includes a metal indenter with tension spring and a scale on which hardness can be directly read. The penetration depth of the sharp point is converted into Barcol values 15 Barcol surface hardness tester is preferred in studies thanks to its portability and direct reading of measurements.[13]
Dental erosion is the pathologic, chronic and localized loss of dental hard tissue as a result of wearing due to a chemical process without the involvement of bacteria.[16-18] The exogenous factors contributing to dental erosion are excessive consumption of acidic fruit juice and acidic carbonated beverages.[19,20] Studies showed that tooth-coloured restorative materials such as GIC, polyacid-modified composite resins and composite resins are also affected by dental erosion.[21,22]
The aim of this study was to examine the effects of different acidic beverages on the surface hardness of commonly used restorative materials.

MATERIAL AND METHODS

The brands, manufacturers and chemical composition of the materials used in this study are listed in Table 1.

Table 1. Composition of the restorative materials used in this study.

Material	Classification	Composition	Shape	Manufacturer	Batch Number
Filtek Silorane	Microhybrid composite resin	Silorane based hydrophobic resin matrix, camphorquinone, yttrium fluoride. Filler weight 76%	A2	3M/ESPE, (St. Paul, MN, USA)	N361058
Filtek Z - 550	Nanohybrid composite resin	Bis-GMA, Bis-EMA, TEGDMA, UDMA. Filler weight 81,8%	A2	3M/ESPE, (St. Paul, MN, USA)	N316660
Filtek Ultimate flowable	Flowable composite resin	Bis-GMA, TEGDMA. Filler weight 65%	A2	3M/ESPE, (St. Paul, MN, USA)	N375701
Dyract Extra	Polyacid-modified composite resin (compomer)	UDMA resin/TCB resin, Strontium-alumino-sodium-fluoro- silicate glass, strontium fluoride, initiators/stabilizers	A2	DENTSPLY (DeTrey, Konstanz, Germany)	1104001712
Fuji II LC	Resin-modified glass-ionomer cement	HEMA, methacrylate, tartaric and polyacrylic acid, fluoroalumino silicate. Filler weight 58%	A2	GC Corporation, (Tokyo, Japan)	1111021

Specimen preparation

For each restorative material, 60 disc-shaped specimens were prepared using a plexiglass mould of eight mm diameter and two mm thickness (a total of 300 specimens). In order to ensure that specimens would have flat polymerized surfaces with no bubble formation after curing, the top and bottom surfaces were covered with a polyester matrix strip (ESR-P Universal strip) and a thin glass microscope slide (one mm thickness). Pressure was applied to extrude excess material.
The restoratives were light polymerized according to the manufacturers’ cure times with LED light curing unit (Elipar FreeLight 2, standard mode; 3M ESPE, St. Paul, MN, USA). With each restorative material, the 60 specimens

were randomly divided into six groups (n=10 per group) according to the six immersion solutions to be examined in this study (Table 2).

Table 2. Study Groups.

Group	Restorative Material	Number of Samples (n)	Sub-Group	Number of Samples (n)
Group 1	Filtek Silorane	n= 60	11- Cola 12- Sour Cherry 13- Apple 14- Energy Drink 15- Orange 16-Artificial Saliva	n=10 n=10 n=10 n=10 n=10 n=10
Group 2	Filtek Z 550	n= 60	21- Cola 22- Sour Cherry 23- Apple 24- Energy Drink 25- Orange 26-Artificial Saliva	n=10 n=10 n=10 n=10 n=10 n=10
Group 3	Filtek Ultimate Flowable	n= 60	31- Cola 32- Sour Cherry 33- Apple 34- Energy Drink 35- Orange 36-Artificial Saliva	n=10 n=10 n=10 n=10 n=10 n=10
Group 4	Dyract Extra	n= 60	41- Cola 42- Sour Cherry 43- Apple 44- Energy Drink 45- Orange 46-Artifical Saliva	n=10 n=10 n=10 n=10 n=10 n=10
Group 5	Fuji II LC	n= 60	51- Cola 52- Sour Cherry 53- Apple 54- Energy Drink 55- Orange 56-Artificial Saliva	n=10 n=10 n=10 n=10 n=10 n=10

Afterwards, they were polished with 600-800-1000-1200-grit silicon carbide paper, as Mathias . (2009) did in their studies. Immediately after polishing, lower surfaces of the specimens were marked with a pen. All specimens were stored in artificial saliva at 37°C for 24 hours for re-hydratation and completion of the polymerization.

Microhardness test

The Barcol impressor consists of a sharp point with a tension spring which is pressed against the surface to be tested. The sharp point has a diamater of 1mm. The value read on the scale decreases as the impressor penetrates the surface.[24] The samples to be tested with this method are placed at the sharp tip of the Barcol Impressor, which is designed to exert a constant load of 10kg. A uniform pressure is exerted on the sample until the scale on the impressor reaches the maximum limit. The scale produces a direct value between 0 and 100.25

After 24 hours, the specimens were rinsed with distilled water and blotted dry with a tissue paper before remeasurement. Surface hardness measurements were conducted with a Barcol surface hardness tester (Sheen, GYZJ 935, Barber Colman Company, U.K.) from three different points on the top surface of each specimen and, average values were calculated.

The surface hardness test was performed twice: Firstly, at the beginning of the study before the specimens were immersed into acidic beverages and secondly, after one month at the end of the study.

Immersion solutions

Ten specimens of each material were immersed in one of the six different solutions (artificial saliva, cola, orange juice, apple juice, energy drink, and sour cherry juice (Table 3).

Table 3. Compositions of acidic beverages and artificial saliva used in the study.

Immersion media (brand)	Manufacturer	Composition	pH
Cola (Coca Cola)	Coca Cola Comp. (USA)	Water, sugar, carbondioxide, colouring agent, cola extract, caffeine, acidity regulator (phosphoric acid)	**2.14**
Sour cherry juice (Cappy)	Coca Cola Comp. (USA)	Water, concentrated sour cherry juice (at least 35%), sugar (sucrose/glucose syrup), acidity regulator (citric acid)	**2.86**
Apple juice (Cappy)	Coca Cola Comp. (USA)	Concentrated apple juice (100%), water, aroma, acidity regulator (citric acid)	**3.14**
Energy drink (RedBull)	RedBull Gmbh (USA)	Water, sucrose, glucose, carbondioxide, taurine, caffeine, inositol, glucuronolactone, vitamins, acidity regulator (sodium citrate)	**3.21**
Orange juice (Cappy)	Coca Cola Comp. (USA)	Concentrated orange juice with real orange pieces (100%) water, aroma	**3.60**
Artificial saliva	Ondokuz Mayis University, Faculty of Science and Letters, Department of Chemistry	1.160 gr/liter (L) Sodium Chloride, 0.600 g/L Calcium Chloride, 0.600 g/L Potassium Phosphate, 1.491g/L Potassium Chloride, 0.050 g/L Sodium Flouride, Trace Sodium Hydroxide to maintain a pH value of 7.	**7.0**

Artificial saliva served as a control solution. The specimens were kept immersed in artificial saliva at 37°C in the interval between cycles.

In this study, immersion media were replaced daily throughout the study in order to prevent pH changes, as did Cogulu *et al* [14] in their studies. The study was planned assuming that individuals consumed these beverages on a daily basis, which was the same procedure adopted by Sarı *et al* [26] The pH values were obtained by using a digital pH electrode (Multi-parameter analyzer, Consort C864, Belgium) which had been calibrated immediately prior to use.

After baseline microhardness was recorded, each sample was first immersed in 32.5 ml of acidic beverage for five seconds and then for another five seconds in artificial saliva. This cycle was repeated ten times daily for each subgroup for a month. The specimen soaking protocol was simulated from an individual drinking a can of soft drink (325 ml). Total soaking time was 100 seconds. After the soaking sequence was completed, the specimen was rinsed with distilled water, blotted dry and subjected to post-immersion microhardness testing. The same procedure was also followed by Wongkhantee *et al.* [21]

Statistical analysis

The "power and sample size" test conducted showed that the sample size was 10 when the confidence interval was 95% (Minitab 13.2 V.).

The data collected in the study were firstly subjected to normality and variance homogeneity tests. To confirm the normality of data distribution, Kolmogrov – Smirnov test was applied. On the other hand, Levene test was used for variance homogeneity. To detect the differences between the materials and liquids, two-way ANOVA test and Tukey multiple comparison test were used.

Paired sample t-test (t-test for dependent samples) was used to reveal the significance of differences between the first and second measurements.

RESULTS

A total of 300 samples were prepared for the study. The pre- and post-experimental surface hardness of the samples was measured with a Barcol surface hardness tester. Table 4 shows the results of the variance analyses conducted on the surface hardness values.

The statistical evaluation of the surface hardness values for Fuji II LC, Filtek Ultimate Flowable, Dyract Extra, Filtek Silorane and Filtek Z 550, which were included in the study, revealed a significant ($p<0.05$) difference among the restorative materials. With regard to the differences among the restorative materials, Filtek Z 550 (96.44 ± 0.13) had the highest hardness value, followed by Filtek Silorane (96.02 ± 0.13) and Dyract Extra (95.98 ± 0.13) respectively. Filtek Ultimate Flowable had a surface hardness of 94.68 ± 0.13. The lowest surface hardness value was measured for Fuji II LC (93.35 ± 0.13). The Tukey and Duncan multiple comparison tests indicated a significant difference among these values.

There were statistical differences among the restorative materials with respect to pre-experimental values and post-experimental values. The classification of the restorative materials with regard to their post-experimental surface hardness values put Filtek Silorane, Filtek Z 550 and Dyract Extra in the same group with the highest surface hardness values, followed by Filtek Ultimate Flowable. Fuji II LC had the lowest surface hardness value (Table 4).

Table 4. Pre- and post-experimental surface hardness values of the restorative materials and statistical relationships among them.

Restorative Material	Pre-experimental mean values (X ± Ss)	Post-experimental mean values (X ± Ss)
Filtek Silorane	96.02 ± 0.13 A	94.76 ± 0.14 a
Filtek Z 550	96.44 ± 0.13 A	94.90 ± 0.14 a
Filtek Ultimate Flowable	94.68 ± 0.13 B	93.78 ± 0.14 b
Dyract Extra	95.98 ± 0.13 A	94.92 ± 0.14 a
Fuji II LC	93.35 ± 0.13 C	92.32 ± 0.14 c

There is no statistical difference between the values represented with the same letter ($p<0.05$)

It was found out that Filtek Z 550 was the most affected restorative material of all. There was no statistically significant difference among the rest of the restorative materials ($p<0.05$) (Table 5).

Table 5. The influence of the beverages on the restorative materials as indicated by the difference between pre- and post-experimental values.

Restorative Material	Difference between pre- and post-experimental values
Filtek Silorane	1.26 ab
Filtek Z 550	1.55 a
Filtek Ultimate Flowable	0.90 b
Dyract Extra	1.06 b
Fuji II LC	1.03 b

There is no statistical difference between the values represented with the same letter ($p<0.05$)

The restorative materials used in the study were soaked in six different liquids. Of these, the lowest difference was produced by artificial saliva in terms of surface hardness compared with the other liquids. This difference was statistically significant ($p<0.05$). As for the acidic beverages, there was no statistically significant difference ($p>0.05$) (Table 6).

Table 6. The comparison of the effects of beverages on surface hardness of restorative materials.

Storage Medium	Change in surface hardness of restorative material
Artificial Saliva	0.5292 **b**
Orange Juice	1.1632 **a**
Energy Drink	1.2220 **a**
Cola	1.2340 **a**
Apple Juice	1.3570 a
Sour Cherry Juice	1.4546 **a**

There is no statistical difference between the values represented with the same letter ($p<0.05$)

DISCUSSION

Many types of food and beverages affect both natural teeth and the restorative materials. Previous studies showed that filling materials, when exposed to low pH media, disintegrated from the resin materials and that matrix components deteriorated. Most of the soft drinks are acidic and their pH values are 3.0 or lower. This suggests that long and continuous sipping of acidic beverages results in an erosion on tooth enamel and resin material.[17,27]

The volume of an acidic beverage or a liquid taken into the mouth is much greater than that of the saliva in the mouth; thus, teeth are exposed to a mixture of saliva and acidic solution for a short time. This lasts for a while and then the erosive liquid is either swallowed or expectorated. Afterwards, the saliva flow cleanses or buffers the acids. Cycles of exposure to saliva are recommended for studies since this approach is the closest to clinical conditions.[28]

The use of saliva in this type of studies is a challenge because the composition of human saliva is complicated and varies by individual oral health.[29] In this study, the same type of artificial saliva used by Francisconi[30], Honorio[31] and De Oliveira[32] was preferred in order to maintain a standard and ideal saliva medium.

It was reported that any food with a pH value lower than the critical 5.5 level might demineralize the hard tissues of the tooth.16 In this study, citric and phosphoric acid containing beverages with pH values lower than 5.5 were used.

One of the most important physical properties of the restorative materials contributing to their clinical success is the surface hardness and it also sets the mechanical properties of the restorative material. It not only increases resistance against scratch and wear but also prevents the easy deformation of the restorative material by various forces, which play an important role in the clinical success.[13,14]

There are different types of surface hardness tests. The most common testing methods used in dentistry for measuring the surface hardness of the restorative materials are Brinell, Rockwell, Shore, Barcol, Knoop and Vickers.[15,33]

Barcol hardness tester (BH) is commonly preferred in studies examining surface hardness due to portability and direct reading of measurements.[34-36] Therefore, BH was used in this study, which was of choice in studies conducted by Bagis and Ertas [37] and Arisu *et al*[38] Previous studies revealed that properties of the filler particles in the matrix such as size, shape, distribution, and content per volume/weight of the filler particles affect the mechanical durability, hardness and flexibility of the composite resins.[39-41]

Similarly, in this study, we found that initial surface hardness value of Filtek Z 550, a nano-hybrid composite resin with the highest filling material ratio, was higher than that of Filtek Silorane, a micro-hybrid composite resin. It was followed by compomer, flowable composite and resin-modified glass ionomer (RMCI) respectively. These results are in compliance with those of Sarı *et al*.[26]

Gonulol *et al*[42], using a standard led device, reported that composite resin had the highest surface hardness value, which was followed by RCMI and compomer. In this study, we found that the surface hardness of compomer and composite resin was statistically same.

In another study conducted by Wassell.[43], surface hardness ranges of vikers (VH) and BH were compared and it was revealed that the range of VH was wider compared to that of BH. It was showed that BH was a reliable but not a sensitive device to compare composites with regard to surface hardness. In our study, the ranges of surface hardness values were close to each other.

Cogulu. [14] used compomer, RMCI and GIC as restorative materials in a similar study. They found that the effects of three different acidic beverages were similar when they evaluated the restorative materials according to the types of beverages. In this study, we found that five different types of acidic beverages had statistically similar effects on the same type of restorative material with respect to surface hardness. We believe that this results from the fact that pH values of the beverages evaluated were close to each other. This complies with the results of Cogulu *et al* . [14]

Ilie *et al*. [44] found that silorane-based composite resin had significantly lower water absorption and solubility values compared to methacrylate-based composites (MBCs) due to its hydrophobic siloxane structure. They also found that it was quite resistant against wear when stored under conditions simulating oral conditions. In this study, we also observed that the decrease in the surface hardness of Filtek Silorane was lower compared to Filtek Z 550, which is an MBCs.

Rios *et al*[45] used GIC, RMCI, composite resin and amalgam in their study on the subject. They soaked these restorative materials in cola for seven days with three cycles lasting five minutes each day. They reported that GIC had the highest values for wear and surface hardness decrease and that composite resin did not have the lowest values for wear and surface hardness change, which was a different finding compared to other studies. They suggested that this resulted from the short experiment period. In this study, we found that composite resin had a higher decrease in surface hardness compared to RMCI. However, this difference was not statistically significant.

Honorio *et al*[31] conducted a similar study using the same GIC, RMCI, composite resin and amalgam as Rios.[45] Their study lasted 35 days and the lowest difference was observed in the restorative material with the highest surface hardness value.

Another study by Bors *et al*[46] involved two composite resins (Filtek Z 550, X-tra fill), two compomers (Dyrac Extra, twinky star) and two glass ionomers (Ketac molar- Fuji II LC) as restorative materials. The samples were soaked in Coca Cola for 30 days with five cycles lasting five minutes each day. The erosive wear of the samples were measured, based on the difference between surface roughness measured at the beginning and the end of the study. It was reported that composite restorative material had a higher erosive wear compared to compomer. This result was attributed to the short experiment period and exposure to cycles.

Badra *et al*. [47] reported that there were several effects on the microhardness and surface roughness of the restorative materials which resulted from the characteristics of the materials, the type of beverage and the evaluation period. They also reported that when the immersion period prolonged, the effects on the properties of the resins were more prominent.

CONCLUSION

1. With the increasing consumption of acidic beverages by the young population in recent years, dental erosion has become an important health issue. We observed that acidic beverages affected the surface hardness of restorative materials used in our study.

2. In our study, all restorative materials, except for Filtek Z 550, underwent statistically similar changes in surface hardness values, which resulted from short study period (a total of 50 minutes of contact with the acidic beverage) and exposure to artificial saliva-beverage cycle.

3. These results show that beverages with erosive potentials affect the surface hardness of restorative materials used in this study. We believe that such studies need to be supported by in vivo studies and that longer storage periods may produce higher statistical differences.

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