

Microhardness And Corrosive Wear of Dental Composite Materials in Alkaline Media

Alkali Ortamda Dental Kompozitlerin Mikrosertlik ve Koroziv Aşınmaları

*Cihat ÇEKİÇ DDS, PhD.

* Associate Professor; Hacettepe University Faculty of Dentistry Department of Prosthodontics

ABSTRACT

Objective: The purpose of this study was to examine the corrosive wear and the microhardness of tooth colored composite resins.

Material and Method: The internal corrosion of dental resin composites is associated with water-sorption. In this study, the internal corrosion and microhardness of packable composites in 0,1 N NaOH were determined and changes compared with those microhybrid resins and ormocers. Internal corrosion in water, artificial saliva and in media of low pH is time consuming, because the degradation process in these environments is extremely slow. In considering this limitation of above media, an alkaline medium has been used in this study to simulate corrosive-wear of 7 composites. For each material namely Solitaire, Surefil, Definite, Admira, Z-100, TPH and Silux Plus, eight cylindrical specimens of 10 mm. diameter and 3 mm thickness were prepared according to manufacturers' instructions. The procedure consists of exposing each material to 0,1 N NaOH at 60°C for 2 weeks. Then the mass change and the microhardness of the materials were determined.

Results: The results indicated statistically significant differences ($p<0.05$) among the tested composite materials. The microhardness values of solitaire, surefil and Z-100 showed a tendency to decrease in relation with time. Definite, Admira, TPH and Silux Plus showed progressive decline in relation to time. It was also observed that TPH showed significantly the lowest mass loss value (0.33%) and solitaire showed the highest (4.63%).

Conclusion: From the results, no one material maybe considered ideal on the basis of its physical properties and characteristics. As a consequence, clinicians should have an informed understanding of the advantages and limitations of alternative material for specific environmental circumstances.

KEYWORDS

Corrosion, Microhardness, Dental composite

ÖZET

Amaç: Bu çalışmanın amacı kompozit rezinlerin mikrosertlik ve koroziv aşınmalarını incelemektir.

Materyal ve Metot: Dental kompozitlerin korozyonu su emilimi ile ilişkilidir. Bu çalışmada 0,1N NaOH ortamında "packable" kompozitlerin mikrosertlikleri ve internal korozyonları incelendi ve mikrohibrit rezinler ve ormocerlerle karşılaştırıldı. Su, yapay tükürük ve düşük pH değerli ortamlarda oluşan internal korozyon çok yavaş ilerler. Bu nedenle bu çalışmada alkali ortam kullanılmıştır. Çalışmada kullanılan kompozit materyallerin (solitaire, surefil, definite, Admira, Z-100, TPH ve Silux Plus) herbirinden 10mm çapta, 3mm kalınlıkta 8 silindirik örnek hazırlandı. Örnekler 0.1N NaOH ortamında, 60°C de 2 hafta bekletildi. Örneklerin kütle değişimleri ve mikrosertlik değerlerindeki değişimler kaydedildi.

Bulgular: Sonuçlar, incelenen materyaller arasında istatistiksel olarak anlamlı ($p<0.05$) farkların bulunduğunu gösterdi. Solitaire, Surefil ve Z-100, zaman içinde azalan mikrosertlik değerleri gösterdiler. Definite, Admira, TPH ve Silux Plus için zaman içinde ilerleyerek azalan değerler kaydedildi.

TPH en az kütle kaybını (%0.33) gösterirken, Solitaire en fazla (%4.63) kütle kaybına uğrayan materyal oldu.

Sonuç: Bulgulara bakıldığında, hiçbir materyalin ideal fiziksel özelliklere sahip olmadığı, klinisyenlerin materyal seçiminde özel çevresel faktörleri de dikkate alarak materyallerin avantaj ve sınırlamalarını gözden geçirmesi gerektiğine inanıyoruz.

ANAHTAR KELİMELER

Korozyon, Mikrosertlik, Dental kompozitler.

INTRODUCTION

The esthetic component of dental care has become increasingly more important, while new tooth colored materials are continually marketed. Recently several manufacturers have introduced “condensable” or “packable” composites to the market with new filler designs that permit them to be packed with more force into cavity preparations as alternatives to amalgam.¹⁻⁵

They are characterized by a high filler load and a filler distribution that gives them a different consistency compared with the hybrid composites.⁶

On the basis of the perceived high-filler load, these materials may be expected to exhibit superior physical and mechanical properties besides the improvements in handling.⁷ One of their limitations, however is their inadequate resistance to wear.^{8,9} It is known that, in any wear phenomenon, four fundamental processes either singly or in combination are involved.¹⁰ Mechanical forces are involved in three of these, namely, abrasive-wear, adhesive-wear, and fatigue-wear.

The fourth, corrosive-wear, results from the conjoint action of chemical and mechanical forces.¹¹ Considering that saliva is potentially aggressive electrolyte, corrosion is likely to play

an important role in the intraoral wear of resin composites.

The hydrolytic degradation (corrosion) is slow in the oral environment.⁽¹¹⁾ For most composite resins, the effects of degradation on wear are not perceptible until a year or longer following the placement of the restoration.

The process is also slow in water and artificial saliva¹²⁻¹⁴ and is even slower in low pH media¹⁵.

It has been noted in a previous study that, of several solvents used in softening of composite resins, the most effective was NaOH solution⁽¹⁶⁾ In the other study, it has been observed that an increase in pH of the medium increases the wear rate of composite resins.¹⁷

The purpose of this study is to examine the corrosive wear and the microhardness of tooth colored composite resins.

MATERIAL AND METHODS

The seven commercial composite restorative materials studied are listed in Table I. In general, the materials consist of a continuous polymeric resin matrix reinforced with a discontinuous filler interfaced to the matrix with silane coupling.⁽¹¹⁾ The filler is either silica or complex silicates or a mixture of two. The fillers in various brands dif-

TABLE I

Materials used in this study

Material	Description	Manufacturer
Solitaire	Packable Composite	Heraus Kulzer, South Bend, IN
Surefil	Packable Composite	Dentsply De Trey, Konstanz, Germany
Definite	Ormocer	Degussa AG, Hanau, Germany
Admira	Ormocer	Voco GmbH, Cuxhaven, Germany
Z-100	Microhybrid Composite	3M, St. Paul, MN, USA
TPH	Microhybrid Composite	Dentsply/Caulk Milford, DE, USA
Silux Plus	Microfil Composite	3M, St. Paul, MN, USA

ferred in composition, concentration and particle size distribution.

The experimental methodology is divided into two parts, corrosive wear and microhardness, to study independently the effects of these variables.

Corrosion test

Light-cured disc specimens (n=8), 10 mm diameter and 3 mm thickness, of each brand were fabricated and final ground with water lubricated 600 grit SiC paper. They were ultrasonically cleaned in distilled water for 1 min and then dried to constant mass at 37 °C. Each specimen was placed in a polyethylene vial containing 20 ml. of 0,1 N NaOH and was stored at 60 °C. At the end of two weeks, the specimens were removed from the solution, washed thoroughly with distilled water and dried at 37 °C for constant mass.¹¹

Resistance to corrosion was measured in terms of mass loss. The mass loss (%) was determined from pre- and post-exposure specimen mass. This parameter represents the solubility of both organic and inorganic components of the materials.

Microhardness test

Circular disks of each material were prepared (3mm thick and 10 mm. diameter) in a stainless steel mold on a celluloid matrix supported with a

glass slab on the top and bottom. The materials were cured according to the manufacturers directions. The materials were final ground with water-lubricated 600 grit SiC paper, washed with distilled water and then exposed to the 0,1 N NaOH at 60 °C for two weeks. Following exposure, the specimens were washed with distilled water.

Microhardness was measured on the top and bottom surfaces with a Buehler Micromet (Buehler, Lake Bluff, IL) equipped with a Vickers indenter at a rate of 50 µm/sec with a load of 25 g. The length of the diagonal of each indentation was measured directly from the graduated eye lens six indentations for every surface were made. The means were calculated and converted to Vickers hardness numbers (VHNs) from cataloged data.

RESULTS

The mean values and standard deviations of microhardness and mass loss of seven restorative materials are presented in Table II, III.

Vickers Hardness

The results of the Vicker's microhardness value for each material with 95% confidence limits are listed in Table II.

The results indicated statistically significant differences ($p < 0,05$) among the tested composite materials.

TABLE II

Mean values and standard deviations of the Vickers hardness of restorative materials

Materials	Time					
	0			2weeks		
	Mean	SD		Mean	SD	P
Solitaire	58.18	1.62		51.85	1.12	0.028*
Surefil	60.05	1.78		54.40	1.79	0.028*
Definite	65.76	1.82		67.48	1.50	0.173
Admira	63.11	2.15		65.51	1.62	0.075
Z-100	56.73	2.34		49.15	2.31	0.028*
TPH	88.36	2.22		92.20	0.97	0.046*
Silux Plus	80.98	1.84		84.50	1.26	0.046*

*Significant ($p < 0.05$)

According to the results of Wilcoxon Signed Ranks Test, there were no significant reduction ($p>0,05$) in surface hardness relative to the initial material hardness of Definite and Admira.

It was observed that Z-100 showed significantly lower initial hardness (56,73 VHN) compared with all other tested materials.

Initial hardness of TPH exhibited a significantly higher value (88,36VHN) than all the other materials tested.

The microhardness values of Solitaire, Surefil and Z-100 showed a tendency to decrease in relation with time. Definite, Admira, TPH and Silux Plus showed progressive decline in relation to time with the ranking of the materials tested being considered to be as follows:

TPH>Silux Plus>Definite>Admira>Surefil>Solitaire>Z-100

Mass Loss

The mean values and standard deviations of the mass loss measurements at varying time intervals are presented in Table III.

For all seven materials, there was a significant reduction ($p<0,05$) in mass loss, relative to the initial mass, following exposure to NaOH media. At 2 weeks, there was a significant difference ($p<0,05$) between the amount of mass loss, following corrosive wear for Solitaire, Surefil and Silux Plus.

The mean and standard deviations of percent mass loss changes between the initial values and the values at two weeks are presented in Table IV.

TABLE III

Mean values and deviations of the mass loss levels of restorative materials (Initial mass 100% 6 specimens of each material).

Materials	Time					
	0			2weeks		
	Mean	SD		Mean	SD	
Solitaire	97.46	1.68		92.95	1.57	0.028*
Surefil	97.26	1.22		94.95	1.40	0.046*
Definite	95.88	1.01		95.28	0.74	0.463
Admira	96.28	1.05		95.40	0.72	0.249
Z-100	96.35	0.82		94.75	1.09	0.075
TPH	96.30	0.93		95.98	1.12	0.753
Silux Plus	97.41	0.68		95.86	1.25	0.046*

*Significant ($p<0.05$)

TABLE IV

Mean values and standard deviations of mass loss changes (%)

Materials	Mass loss due to wear %		n=6
	Mean	SD	
Solitaire	4.63	1.39	
Surefil	2.38	1.34	
Definite	0.61	1.71	
Admira	0.87	1.60	
Z-100	1.65	1.92	
TPH	0.33	1.12	
Silux Plus	1.59	1.26	

Chi-square = 18.731
P = 0.005

*Significant ($p<0.05$)

The percent increase in wear due to corrosion were examined by Kruskal-Wallis test and it was found significant differences among all the materials tested. ($p < 0,05$)

It was observed that TPH showed significantly the lowest mass loss value (0,33%) and Solitaire showed the highest (4,63%). The materials tested could be ranked as follows:

Solitaire>Surefil>Z-100>SiluxPlus>Admira>Definite>TPH

DISCUSSION

The progress in developing wear resistant resin composites has been rather slow mainly because wear evaluation has been dependent on time consuming clinical trials. Accelerated laboratory tests are available.²⁰⁻²³ While these tests have been useful in studying fundamental mechanisms of wear, they have not been able to predict clinical wear.²⁴⁻²⁶ One of possible reason for this difficulty is that, environmental degradation has not been taken into consideration in the design of most in vitro wear tests.¹¹

It is recognized that the alkaline medium used in this study does not represent the composition of saliva nor its pH. The pH of the storage solution affects degradation of composite resin materials.^{27,28} This effect has been discussed in terms of matrix decomposition which occur due to hydrolysis of the polymer matrix.¹⁹ The degradation process has been shown to associated with the swelling causes the formation of pores inside the material from which organic substances can be released, resulting in a mass loss.¹¹⁻¹⁹ Wear is enhanced by loss of reinforcement resulting from the hydrolysis of silane coupling and filler particles Neither water nor alcholic solvent is capable of enhancing this corrosion process that preceedes wear.¹¹ Chadwick et. al.²⁹ reported that a low pH of the storage solution has a negative effect on the wear resistance of composite resins. A high pH is detrimental to the stability of silane coupling and enhances dissolution of both silica and silicate glass. For a specific filler composition, their particles dissolve faster than coarser particles.¹¹ With respect to inorganic fillers their composition, content and particle size distribution are important variables in internal corrosion. Glass fillers are more hydrolytically unstable than fillers containing silica.

A suitable medium for in vitro corrosive-wear testing is one that accelerates the hydrolysis process. Since this process involves interaction with the OH^- ion, a medium with an excess of hydroxyl ions can serve this purpose. Based on this rationale, we have used an aqueous 0,1 N NaOH solution in studying the corrosive-wear of resin composites.¹¹ Exposure of composite resin to this medium for a few hours produces surface microstructural damage similar to that observed in restorations retrieved from clinical service.

As shown by the result of the present study, mass change of the different composites was affected with 0,1 N NaOH solution. Of all the materials tested TPH proved most resistant to corrosion and mass loss (0,33%). Conversely, Solitaire showed the highest mass loss value. (4,63%) For comparing the corrosive wear of the materials, TPH performed the best and Solitaire the worst. Subsequently, there was reduction in mass, so that after 2 weeks, all had become lighter by an extent compared with the initial values. That was statistically significant for Solitaire, Surefil and Silux Plus. ($p < 0,05$)

The corrosive wear rate of composites has been reported to be related to the amount, size, and degree of filling particles in the matrix.¹⁹

Within the limits of this study, negative correlation was observed between hardness and corrosion of the composite resins.

The hardness of a material is the resistance a solid shows to local deformation.³⁰ A positive correlation has been established between the hardness and the organic filler content of dental restorative composites.³⁰ Hardness is also dependent on the degree of polymerization. In the present study TPH was found to be the hardest material. Microhardness value of Definite and Admira were higher than Surefil but lower than Silux Plus.

All materials tested showed a tendency to decrease in relation to time. From the findings of the present study, there is no obviously superior material, because time dependent different rankings were obtained from different tests.

It is evident that the chemistry of the oral environment must be taken into account, although the effects differ according to the type of material. Further work is necessary to determine whether the physico-chemical effect can occur

under clinical conditions. These results can be used as a reference for future clinical observations and comparisons.

CONCLUSIONS

The results showed the corrosive wear rate and microhardness of composite resins. TPH showed the least wear rate and the highest microhardness value.

From the results, no one material may be considered ideal on the basis of its physical properties and characteristics. As a consequence, clinicians should have an informed understanding of the advantages and limitations of alternative materials for specific environmental circumstances.

REFERENCES

- Leinfelder KF: New development in resin restorative systems. J. Am. Dent. Assoc.1997; 128: 573-581
- Leinfelder KFRadz GM, Nash RW: A report on a new condensable composite resin. Compendium 1998; 19: 230-237
- Leinfelder KF, Prasad A: A new condensable composite resin for the restoration of posterior teeth. Dent. Today 1998; 17:112-116
- Adams TC: A high-density posterior composite used for an amalgam replacement. Dent. Today, 1999; 18:74-77
- Meier JC, Kazemi R, Meier CD: Microleakage of packable composite resins. Oper.Dent. 2001; 26: 121-126
- Manhart J, Kunzelmann KH, Chen HY, Hickel R: Mechanical properties and wear behavior of light -cured packable composite resins. Dent Mater. 2000; 16: 33-40
- Manhart J, Kunzelmann KH, Chen HY, Hickel R: Mechanical properties of new composite materials. J. Biomed Mater. Res. 2000; 53: 353-361
- Roulet JF: The problems associated with substituting composite resins for amalgam. A status report on posterior composites. J. Dent. 1988; 16: 101-113
- Swift EJ Jr: Wear of composite resins in permanent posterior teeth. J. Am. Dent. Assoc. 1987; 115: 584-588
- Kalachandra S: Influence of fillers on the water sorption of composites. Dent. Mater. 1989; 5: 283-288
- Sarkar NK. Internal corrosion in dental composite wear: Its significance and simulation. J. Biomed.Mater. Res. 2000; 53: 371-380
- Solderholm KJM, Mukherjee R, Longmate j: Filler leachability of composites stored in distilled water or artificial saliva. J. Dent. Res. 1996; 75: 1692-1699
- Pilliar RM, Vowles R, Williams DF: The effect of environmental aging on the fracture toughness of dental composites J. Dent. Res. 1987; 66:722-726
- Ferracane JL, Hopkin JK, Condon JR: Propeties of heat-treated composites after aging in water. Dent Mater. 1995; 11:354-358
- Kond S, Ohkawa S, Hanawa T, Sugawara T, Ota M: Environmental durability of composite resins in acidic and alkaline solutions J. Dent.Res. 1989; 68: 447-922
- Mante F, Saleh N, Mante M: Softening patterns of post-cure heat treated dental composites. Dent. Mater. 1993; 9:325-331
- Wendt SL, de Gee AJ, Pallav P, Werner A, Davidson CL: In vitro wear tested at neutral and acidic conditions. J Dent. Res. 1994; 73:38-106
- Tanoue N, Matsumura H, Atsuta M: Wear and surface roughness of current prosthetic composites after toothbrush/dentifrice abrasion. J.Prosthet Dent. 2000; 84:93-97
- Demirel F, Saygılı G, Şahmalı S: Comparative mechanical property characterization of three indirect composite resin materials compared with two direct composites. Polym. Adv. Technol. 2003; 14:380-386
- Leinfelder KF, Beaudreau RW, Mazer RB: An invitro device for predicting clinical wear. Quint Int 1989; 20:755-761
- Condon JR, Ferracane JL: Evaluation of composite wear with a new multi-mode oral wear stimulator. Dent Mater 1996; 12:218-226
- Ehrnford L, Derand T, Larson LA, Svensson S: An abrasion test for composite resins. J Dent Res 1980; 59:716-720
- Lutz F, Phillips RW, Roulet JF, Setcor JC: In vivo and in vitro wear potential posterior composites. J Dent Res 1984; 63:914-920
- Pearson GJ: Long term water sorption and solubility of composite filling materials J Dent 1979; 7: 64-68
- Kalachandra S, Wilson TW: Water sorption and mechanical properties of light-cured proprietary composite tooth restorative materials. Biomater 1992; 13:105-109
- Wu W, Toth EE, Moffa JF, Ellison JA: Subsurface damage layer of in vivo worn dental composite restorations. J Dent Res, 1984; 63:675-680
- Göpferich A: Mechanisms of polymer degradation and erosion. Biomaterials 1996; 17:103-114
- Geurtzen W: Substances released from dental resin composites and glass-ionomer cements. Eur.J.Oral Sci. 1998; 106:687-695
- Chadwick RG, Mc Cabe JF, Walls AWG, Storer R: The effect of storage media upon the surface microhardness and abrasion resistance of three composites. Dent Mater. 1990; 6: 123-128
- Saygılı G, Şahmalı S, Demirel F: Changes in the mechanical properties of tooth-colored direct restorative materials in relation to time. Polym.Adv. Technol.2003;14:616-622

CORRESPONDING ADDRESS

Cihat ÇEKİÇ DDS, PhD

Hacettepe University, Faculty of Dentistry, Department of Prosthodontics, 06100 Sıhhiye - Ankara / Türkiye
Fax: 90-312-311 37 41