Influence of aging solutions on wear resistance and hardness of selected resin-based dental composites

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Abstract:

Purpose

The purpose of this study was to investigate the effect of different plasticizing aging solutions on wear resistance and hardness of selected universal resin-based dental composites.

Methods

Three light cured (one nanofilled, two microhybride) and one hybride chemical cured composites were aged at 37°C for 48 h in distillated water, ethyl alcohol solution or Listerine mouthwash. After aging the microhardness tests were carried out and then tribological tests were performed in the presence of aging solution at 37°C. During wear testing coefficients of friction were determined. The maximal vertical loss in micrometers was determined with profilometer.

Results

Aging in all liquids resulted in a significant decrease in hardness of the tested materials, with the largest values obtained successively in ethanol solution, mouthwash and water. The effect of the liquid was dependent on the particular material, but not the type of material (interpreted as the size of filler used). Introduction of mouthwash instead of water or ethanol solution resulted in a significant reduction in the coefficient of friction. The lowest wear resistance was registered after aging in ethanol and for the chemical cured hybrid composite, but the vertical loss was strongly material depended.

Conclusions

The effect of different aging solution, including commercial mouthrinse, on hardness and wear was material dependent, and cannot be deduced from their category or filler loading. There is no simple correlation between hardness of resin-based dental composites and their wear resistance, but softening of particular composites materials during aging leads to the reduction of its wear resistance.

Keywords: Properties, microhardness, dental composite, friction and wear tests, aging solution

1. Introduction

Dental amalgams and composites are popular direct restorative materials in clinical practice. Advantages and disadvantages of these materials were widely discussed in the past, but today dental resin-based composites are the most frequently used restorative materials. This materials are used to replace and restore lost dental tissue or to cement fixed dental prosthesis. Resin-based composite is usually composed of polymeric matrix reinforced with filler particles bounded to the matrix by coupling agents. Resin-based composites have a higher level of aesthetics as compared to the amalgam due to the shade similar to the color of natural teeth. Composites, due to the possibility of obtaining materials with varied viscosity, may also be easy manipulated and molded before curing.. Additionally, dental composites eliminate the possibilities of leakage of mercury and waste disposal. However, in the case of composite friction and leaching, components of the polymeric matrix, as well as micro - and nanofillers may enter the body by swallowing and inhaling and accumulate in the tissues potentially affecting the liver, kidneys and intestine [\[\[9\]\]](#page-10-0)-[\[\[10\]\]](#page-10-1). Anyway, the quantities of worn composite material ingredients are rather low and probably do not reach levels that can increase risks of toxic or mutagenic effects [\[\[14\]\]](#page-11-0), but there is still a need for long-term clinical investigations. Wear of composite restorations has no consequences for the temporomandibular joint or periodontium [\[\[13\]\]](#page-10-2). Heintze [\[\[14\]\]](#page-11-0) notes that in fact there is no scientific evidence that higher wear of restorative materials is related to side effects and concludes that the wear is mainly an aesthetic problem, although it probably can restrict some masticatory functions. Preferably, the wear of a dental restorations should be comparable to that of enamel, nonetheless for composite materials it is still higher, althoughlower than some years ago [\[\[2\]\]](#page-10-3). Clinically excessive wear of the restorations is observed usually in patients with bruxism [\[\[11\]\]](#page-10-4). Generally, the wear resistance of molar restorations is lower than that noted for premolar restorations [\[\[21\]\]](#page-11-1). Some modern composites present also similar wear resistance to amalgams [\[\[16\]\]](#page-11-2). So far it is not clear which of the many factors play the most important role in their resistance to friction (degree of polymerization, filler debonding or filler degradation) [\[\[21\]\]](#page-11-1). Although wear resistant is still considered as an important factor, the determination of the longevity of this type of material, especially in large direct restorations or in patients with oral habits needs further investigations [\[\[16\]\]](#page-11-2). The enzymes and ethyl alcohol present in the oral cavity may also influence the degradation of the composite matrix [\[\[17\]\]](#page-11-3).

The aim of this study was to investigate the impact of applying plasticizing aging solutions, such as distilled water, ethyl alcohol and liquid mouthwash containing alcohol on wear resistance and hardness of four widely used dental composites. Now it is: The hypotheses was that wear resistance and hardness is particular material dependent and show no general relation with materials type (interpreted fillers size used and curing method).. Additionally, we tested hypothesis that there is no general relationship between hardness and wear resistance of resin-based dental composites.

2. Materials and Methods

2.1. Materials and samples preparation

We used four widely used composite materials for direct fillings: three light and one chemically cured (Table 1). Test specimens measuring $25 \times 7 \times 1.8$ mm were polymerized in accordance with the manufacturers' instructions in a mould made of stainless steel. For light curing materials lamp Denjoy DY400-4 (Denjoy Dental China, China) was used. There were twenty samples made of each material - five intended for initial microhardness measurements and fifteen for microhardness measurements and wear tests after aging in different solutions. Cured samples were first wet-grounded on abrasive papers (Struers A/S: Copenhagen, Denmark) with the grit size sequence 500 and 1200 on grinding and polishing machine (LaboPol-25, Struers A/S: Copenhagen, Denmark). The samples were thoroughly rinsed with water, and next their working surfaces were polished with 6 μ m, 3 μ m and finally 1 μ m diamond paste (Struers A/S: Copenhagen, Denmark). After polishing, the samples were placed in distilled water in an ultrasonic cleaner for 4 min.

Then, fivesamples of each material were placed in distilled water, five in ethyl alcohol (Avantor, Gliwice, Poland) solution in distilled water (75% vol.) and five in mouthwash Listerine Ultraclean Arctic Mint (Johnson & Johnson, Poland). All samples were stored for 48 \pm 1 h at 37°C \pm 1°C. Additionally five samples of each material were placed in distilled water for 2 h at 37° C \pm 1°C for measurement of the initial microhardness.

2.2. Vickers hardness tests

Samples for initial measurements and after aging were dried from visible moisture using filter paper and next were air-dried for approximately 1 minute. The changes in Vicker's hardness were evaluated using a microhardness tests (FM-7 Future Tech, Tokyo, Japan) with a 300 g load and all loading times were 15 s. Measurements were made 15 times at randomly chosen locations on 5 specimens for each solution (3 measurements for each specimen).

2.3. Wear test

Tribological tests were carried out using CSM Tribometer (CSM Instruments, Peseux, Switzerland). The samples stored in aging liquids were placed in the holders of the environmental chamber made for the purpose of the experiment (Fig. 1). The system was mounted in a tribometer and placed at the upright position. The chamber equipped in a heater with a thermostat was filled with an aging solution (the same solution which was used for further testing). The test the temperature of the aging liquid was $37 \pm 1^{\circ}$ C. During wear testing the specimens were kept in permanent contact to the spherical antagonist $(A₁₂O₃$ ball, 5,5 mm in diameter). The handle of the tribometer was moving together with the minichamber back and forth. For one cycle a linear sliding distance was 8 mm (back-and-forthmovement, 2×4 mm) and the speed of the specimen's movement was 40 mm/s. The vertical load was 50 N. For each sample 10000 full cycles were performed.

While wear testing coefficients of friction were determined, and the three values (after 3000, 6000 and 9000) were taken for each specimen for each aging condition (15 measurement for each material in each condition).

After wear tests specimens were carefully moved using the tweezers to a desiccator containing freshly dried silica gel and put in the dryer at temperature of $45\pm1\degree C$ for 10 minutes to remove moisture from the surface. After drying samples were removed from desiccator and fixed using double-sided adhesive tape to the stainless steel plate. The maximal vertical loss in micrometers was determined with Surtronic 25 (Taylor-Hobson, Leicester, United Kongdom) profilometer. At each sample, the measurements were made at three locations in a perpendicular direction to the moving direction of the sample: first measurement in the middle of the track and two next wipes approx. 1 mm from the center of the trace. For each material – aging condition combination 15 measurements were taken.

2.4. Statistical analysis

The results were subjected to statistical analysis with Statistica 10 software. The distributions of the residuals were tested with Shapiro –Wilk test and the equality of variances was tested with Levene test. When the distribution of the residuals was normal and the variances were equal, the one-way ANOVA with Tukey HSD *post-hoc* tests were used $(\alpha=0.05)$. As the distributions of the residuals were not normal and/or the variances were not equal, the nonparametric Kruskal-Wallis test (α =0.05) was used. When the null hypothesis was rejected, multiple comparisons of mean ranks for all groups were conducted with an aid of a post-hoc test (α =0.05). The correlation of Vickers microhardness and vertical wear was tested by linear regression analysis (α =0.05).

3. Results

Changes in average microhardness of the tested materials are shown in Fig. 2. Before aging, the highest hardness was recorded for the FSU, and the lowest for the CHA. Before aging and in each of the aging solution used different materials showed statistically significant differences in microhardness results $(P \le 0.05)$. A detailed summary of the results of statistical tests is presented in Table 2. The post-hoc test showed no statistically significant differences between microhardness FSU and HER stored in distilled water and after aging in ES and LI between the CHA and BL. The microhardness of each of the test materials affected $(p \le 0.05)$ the type of the aging solution (Table 3). The aging of samples in the ES caused the largest percentage drop in hardness (Table 4). In the case of FSU there was no statically significant difference between the hardness of the aged samples DW and liquid mouthwash, and in the case of materials CHA and BL there were no statistically significant differences in the hardness of the aged samples ES and LI.

The values of the friction coefficients are shown in Fig. 3. The type of the material does not show a statistically significant effect on the friction coefficient $(P> 0.05)$. Type of the solution in which the tribological tests were carried out substantially differentiated coefficients of friction (Table 5), due to lower values than twice the coefficient of friction for the test run in the LI than in DW and ES. There was no statistically significant difference between friction coefficients when using DW and ES.

The results of measurements of vertical wear are shown in Fig. 4. The materials aged in various liquids materials exhibit differences in vertical wear. For FSU and BL materials, there were no statistically significant differences in vertical wear after aging in distilled water and for HER and CHA materials after aging in LI (Table 6). The type of the aging solution showed a significant effect on the obtained values of the vertical wear for each of the materials (Table 7). For three of the four materials there was a decreased in vertical wear after aging in both liquids containing ethyl alcohol. As compared to the results obtained after aging in water, samples after aging ES vertical wear increased by between 38.5% to 65.6%, and after aging in LI the vertical wear increased from 12.1 to 30.2%, but for FSU dropped by 31.8 % (Table 8). HER turned out to be the most wear-resistant material.

The results of regression analyses did not show general relationships between Vickers microhardness and vertical wear when results for all materials and all aging solutions were

analyzed together ($R^2 = 0.0433$, P=0.1107). Similar results were obtained when relationships for all materials in particular aging solutions were analyzed (in distilled water $R^2 = 0.0052$, P=0.7623; in ethanol solution $R^2 = 0.2154$, P=0.0393; in mouthrinse $R^2 = 0.1987$, P=0.0489). Anyway, for particular materials stored in different solutions analyses show correlation between surface hardness and vertical wear (for FSU $R^2 = 0.406$, P=0.0106; for HER $R^2 =$ 0.8497, P<0.0001; for CHA $R^2 = 0.4473$, P=0.0064 and for BL $R^2 = 0.4899$, P=0.0037) and increasing of vertical wear was associated with the hardness reduction.

4. Discussion

Investigated resin-based composites are materials for direct restorations in posterior and anterior teeth, called commonly "universal composites. It is known that for anterior restorations the aesthetic appearance (including polishability) is important, so it usually have small filler particles to increase smoothness, but this also can reduce some mechanical properties, when for posterior restorations wear resistance and high fracture strength are considered as the most important properties [\[\[1\]\]](#page-10-5). Universal composites should combine good polishability and mechanical properties and their use is still growing. The physical and chemical processes during aging have a strong effect on the degradation of the composites in the oral cavity and laboratory test, with reduced longevity and mechanical properties of those materials [\[\[15\]\]](#page-11-4). Thus, in the presented study the influence of different aging solutions, including popular antiseptic mouthwash, on chosen properties of universal composites reinforced with different filler types were investigated.

The microhardness tests are widely used and accepted reliable methods to evaluate and compare the composite resins which may suggest the pre-selection of materials [\[\[6\]\]](#page-10-6), [\[\[24\]\]](#page-11-5). Generally increased filler levels resulted in higher initial microhardness values, which is consistent with the results of other laboratory investigations [\[\[18\]\]](#page-11-6), [\[\[22\]\]](#page-11-7), but hybrid chemically cured BL presented significant lower hardness than nanofilled FSU and microhybrid HER despite the highest filler content (82 % by mass). One of the reasons for the lower HV values of BL might be the relatively low conversion rate of carbon double bonds [\[\[3\]\]](#page-10-7), [\[\[23\]\]](#page-11-8), but this supposition for those material needs verification. The lowest initial surface hardness values for CHA with the lowest filler content can be connected to the results of long-term clinical study by Da Rosa Rodolpho et al. [\[\[5\]\]](#page-10-8), where those materials have shown shorter survival rate, especially in posterior teeth. Some works have shown that hybrid composites present a higher microhardness than nanoparticle composites [\[\[20\]\]](#page-11-9), [\[\[28\]\]](#page-12-0). However other studies proved that nanofilled resin composites show mechanical properties similar or even better than these of universal hybrids composites [\[\[4\]\]](#page-10-9), which is in accordance with presented results. Tornavoi et al. [\[\[22\]\]](#page-11-7) suggests that smaller-sized particles can be more favorable to the mechanical properties when particles show good distribution because the distance between particles becomes reduced, so the contact area increases. This condition justifies the obtained results because FSU with the highest surface hardness values is filled only by nanoscale particles and contains the same filler content by mass like microhybrid HER. After aging all materials in all solutions show lower surface hardness values, what corresponds well with other studies [\[\[28\]\]](#page-12-0), [\[\[19\]\]](#page-11-10). As expected, the highest reduction of microhardness was obtained successively after aging in ethanol solution, next in mouthwash containing 21.6% of ethanol and distilled water. The same as alcohol, water acts as a solvent on the composite polymer matrix, which has been described as plasticizing effect. The polymer chains are separated by a molecules that doesn't form primary chemical bonds with the chain. Instead of it molecules serves as a space occupier and reduce chain interactions, like secondary bonding and entanglements [\[\[7\]\]](#page-10-10). For this reason, hardness and reduction of other mechanical properties is related to plasticizers' uptake. Aging solutions of 50–75% ethyl alcohol in water have been proven to be the most effective plasticizer for dental polymer matrix [\[\[27\]\]](#page-12-1). Reduction of surface hardness of dental composites stored in mouthrinse was also shown by Yap et al. [\[\[29\]\]](#page-12-2) and Festuccia et al. [\[\[8\]\]](#page-10-11). Our results also correspond to Schwartz et al. [\[\[19\]\]](#page-11-10) investigations, who showed the highest hardness reduction after aging in 25% ethanol–water solution for composite with the smallest size of the particles. Schmidt et al. [\[\[18\]\]](#page-11-6) suggested that when smaller filler particles were used, diffusion into deeper layers occurs more slowly. Contrary to Schwartz et al. [\[\[19\]\]](#page-11-10) obtained greater percentage hardness reduction (Table 4) for nanofilled material in distilled water like for microhybrid light cured materials. It indicates that for commercially available restorative composites the relation between the filler size and surface hardness reduction is not obvious. Hardness reduction may be also matrix composition and filler type dependent.

The investigations have shown that after introducing of aging solution as a lubricant between two body we obtained similar coefficient of friction for water and ethanol solution, and significant reduction (almost two and a half times) of coefficient of friction after use of mouthrinse. The effect of mouthrinses on coefficient of friction has not been reported in the literature. Reduced friction coefficient obtained after the application of mouthrinse may be a result of the presence of oils: thymol, eucalyptol, menthol, which are dissolved in alcohol. Essential-oils in mouthrinse composition are used as antiplaque potential of multiple

antimicrobial agents [\[\[26\]\]](#page-11-11), and registered coefficient of friction reduction is rather an additional result.

A friction force has greater effect on rough than on smooth surfaces [\[\[13\]\]](#page-10-2), so in this study we used the same protocols to standardize the surface of the samples before the tests. In our study microhybride HER showed the best compilation of vertical wear after aging in different solutions, and hybride BL presented the worst wear resistance.

In distilled water nanofilled material showed higher vertical wear than both microhydride composites, and similar to hybride BL. After aging in ethanol solution used as the most destructive medium [\[\[27\]\]](#page-12-1), chemical-cured BL presented definitely the highest vertical wear, and the microhydride composites still shown the most favorable wear resistance. In other research works the results for the different types of the composites remain controversial. Turssi et al. [\[\[25\]\]](#page-11-12) showed that microfilled composite have decreased wear resistance in comparison to the nanofiled material. Yesil et al. [\[\[30\]\]](#page-12-3) found similar wear resistance for nanofilled and microfilled materials. Lazaridou et al. [\[\[16\]\]](#page-11-2) showed that the majority of modern resin composites presented good wear resistance, but their results proved that some nanofilled materials were better than microfilled, and vice versa. In our study the wear resistance for two microhybride composites was different, because HER exhibited lower vertical wear than CHA which has the lowest filler loading (Table 1). On the other hand, the hybrid material BL with the highest filler loading shows the lowest wear resistance and simultaneously nanofilled composite shows better properties than BL, but generally worse than CHA. This result corresponds well to the other findings obtained for today used materials, where relationships between wear resistance and filler content (wt%) are rather weak, if any exist [\[\[12\]\]](#page-10-12), [\[\[16\]\]](#page-11-2). After aging in mouthrinse significant changes were noted. Effect of mouthrinses on composites wear has been studied so far only by Yap et al. [\[\[29\]\]](#page-12-2). Our results show that the vertical wear in mouthrinse for microhybrid and hybrid composites was higher than in water, but lower than in ethanol solution. This was in line with expectations resulting from Yap et al. [\[\[29\]\]](#page-12-2) findings. However nanofilled materials aged in Listerine showed reduced vertical wear in comparison to the results obtained after aging in distilled water and ethanol solution. It can be supposed that reduction of coefficient of friction and thin lubricant layer formation on samples by mouthrinse fulfill the protective role by limiting the number of nanoparticles removed from the polymer matrix in more effective way than for composites with larger particles, but this speculation need further investigations. However it should be noted, , that in real conditions there is no constant contact of the mouthrinse with restoration that was during laboratory tests. Even if the potential protective

role of mouthrinse for nanocomposites will be confirmed in further studies, in practice, this role will be limited in time because the produced film during chewing will be worn and / or flush by the saliva from the fillings.

The results of regression analyses show that there was no simple relations between surface hardness and wear resistance of dental composites, so some harder materials may be worn faster. However, reduction of hardness after aging is related with decrease wear resistance of particular materials.

The considerations shown above prove that there is no direct correlation between the filler loading or the fillers size (composite type) and vertical wear or hardness in different aging media. Modern composite resins differ in filler size and concentration, particle type interaction, such as quartz or silica, fillers distribution, morphology, chemical composition (type of organic matrix, initiator) or degree of conversion. This creates a large variation in composite properties. According to the presented results and with limitations related with number of tested materials, the wear resistance of composite resins is material-dependent, and it is very difficult to determine the wear resistance of materials according to their type (microhybride, nanofilled) and filler loading.

5. Conclusions

Within the limits of this study, the following conclusions were formulated:

- The effect of different aging solutions, including commercial mouthrinse, on hardness and wear was material dependent, and cannot be deduced from their category or filler loading.
- There is no simple correlation between hardness of resin-based dental composites and the wear resistance, but softening of particular composite material during aging leads to the reduction of its wear resistance.
- Mouthrinse used as a lubricant solution in wear tests significantly decreased coefficients of friction.
- Within the limitation of the presented laboratory tests, for nanofilled material softening in mouthrinse that contain alcohol was not the major factor determining wear resistance, probably due to the protective role of lubricant properties of aging solution, but this phenomena need further verification for other nanofilled materials.

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Fig. 1. Tribometer with the environmental mini-chamber attached.

Figures:

Fig. 2. Vickers microhardness of four composite restorative materials stored in different aging solutions.

Fig. 3. Coefficient of friction registered during wear test in different aging solutions

Fig. 4. Vertical wear of four composite restorative materials stored in different aging solutions.

Tables:

Table 1. The chemical composition of materials used in the tests according to the manufacturers' informations.

Table 2. Comparison of Vickers hardness for various restorative materials stored in different aging solutions; one-way ANOVA and Tukey post-hoc test results, $\alpha = 0.05$.

FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light

Table 3. Comparison of Vickers hardness for particular material's stored in different aging solutions; one-way ANOVA and Tukey HSD post-hoc test results, $\alpha=0.05$.

Aging solution (I) group	solution Aging (J) group	P-values			
		FSU	HER	CHA	BL
		(<0.0001)	(<0.0001)	(<0.0001)	(<0.0001)
BA	DW	0.0002	0.0015	0.0013	0.0002
	ES	0.0002	0.0002	0.0002	0.0002
	LI	0.0002	0.0002	0.0002	0.0002
DW	ES	0.0002	0.0002	0.0002	0.0002
	LI	0.0748	0.0002	0.0002	0.0002
ES	LI	0.001	0.0008	0.6713	0.4093

BA - before aging, DW - distilled water, ES - ethanol solution, LI – Listerine, FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light

Table 4. Percentage hardness changes of four restorative materials during aging in different liquids.

Aging	Hardness reduction, %.					
solution	FSU	HER	CHA	BL		
DW	11,5	6,7	6,7	12,1		
ES	23,5	42.4	26,2	29,4		
	16,2	35,3	24,8	27,0		

DW - distilled water, ES - ethanol solution, LI – Listerine, FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light

Table 5. Comparison of coefficient of friction for particular material's stored in different aging solutions; Kruskal-Wallis ANOVA test and multiple comparisons of mean ranks for all groups post-hoc test results, α =0.05.

DW - distilled water, ES - ethanol solution, LI – Listerine, FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light

Table 6. Comparison of vertical wear of composite restorative materials stored in different aging solutions; one-way ANOVA and Tukey post-hoc test results, $\alpha = 0.05$.

Restorative material (I) group	Restorative material (J) group	P-values			
		Distillated water	Ethanol solution	Listerine (<0.0001)	
		(<0.0001)	(<0.0001)		
FSU	HER	0.0002	0.0002	0.0004	
	CHA	0.0002	0.0002	0.0002	
	BL	0.4081	0.0002	0.0002	
HER	CHA	0.0002	0.0002	0.0597	
	BL	0.0002	0.0002	0.0002	
CHA	BL	0.0002	0.0002	0.0002	

FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light

Table 7. Comparison of Vertical wear, um for particular material's stored in different aging solutions; one-way ANOVA and HSD Tukey *post-hoc* test results, α=0.05.

DW - distilled water, ES - ethanol solution, LI – Listerine, FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light

Table 8. Percentage vertical wear changes of four restorative materials during aging in different liquids (reference point was aging in distilled water).

ES - ethanol solution, LI – Listerine, FSU - Filitek Supreme Ultra, HER - Herculite XRV, CHA - Charisma, BL - Bright Light