

Effect of Mica as Filler on Wear of Denture Teeth Polymethylmethacrylate (PMMA) Resin

SUMMARY

The purpose of the study was to determine the effect of mica as filler on in vitro wear of denture teeth PMMA resin. Denture teeth PMMA resin was modified with 4 percentages (5, 10, 15, 20% by weight), silanized and unsilanized mica filler as the test group specimens. Control specimens were unfilled. All of specimens were tested at initial and after 90 days with 2-body wear testing device. Mann-Whitney U, Wilcoxon and Kruskal Wallis 1-Way Anova tests were used for the statistical analysis ($p < 0.05$). Test specimens with silanized mica filler displayed a weight loss significantly lower, whereas test specimens containing unsilanized mica lost significantly more weight ($p \leq 0.05$). With the exception of the 20% group ($p = 0.07$), silanization increased wear resistance significantly ($p \leq 0.01$). With increasing mica concentration, total weight loss of the silanized group did not show a significance change ($p > 0.05$); in the unsilanized groups, the weight loss fitted a variable distribution pattern ($p \leq 0.01$). The wear of all test specimens, except for unsilanized 20% ($p = 0.02$) and silanized 10% ($p = 0.007$) groups, was not affected negatively after stored at open atmosphere. The percentage of 5% silanized mica filler was found to give the best wear resistance results, but unsilanized mica was found to weaken the denture teeth PMMA resin.

Keywords: Wear; Mica; Denture Teeth; PMMA Resin

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Introduction

In relation to the long-term conservation of aesthetic, the wear of artificial teeth is important due to their influence on the maintenance of maximum intercuspitation at centric jaw relation position, masticatory efficiency, the occlusal vertical dimension, and occlusal stability. For this reason choosing the artificial teeth material is very important. Artificial teeth replacements are made from 3 types of material: porcelain, acrylic resin and modified resin. Acrylic resin teeth, which have been widely used in clinic, have some advantages over porcelain teeth: acrylic resin teeth have excellent fracture toughness, easy occlusal adjustment and high bond strength to denture base materials, but their wear resistance has been questioned^{6,10,20}. Recently, composite materials have received some interest as denture teeth material⁶. New

artificial teeth (composite resin) have been developed for improving their mechanical properties and wear resistance. A composite with filler particles and cross-linked polymers are used as the material for artificial teeth^{12,24}. Inorganic fillers in the composite teeth are subjected to a silane-coupling treatment to enhance adhesion of these particles to the matrix base of the teeth material^{4,9,14,18,21,28,31}.

It is believed that the greatest improvement in abrasion resistance of especially resin composites can be maintained by using solid, high Mohs, broad size distribution filler having strong bond/matrix interaction *via* surface modification to maximize bonding²². It is very well known that compared with acrylic resin denture teeth, isosit denture teeth containing silica filler wear less^{17,28,31}. The addition of quartz in various ratios to polymer that is used in the production of artificial teeth has been reported to diminish the wear significantly^{29,30}. Among the other reinforcing fillers, mica has silica sheet structure with

biotite and muscovite, which is mainly aluminium silicate and exhibits an intermediate Mohs hardness (2.5-3)²². It has been used as filler for plastics due to its low cost, easy availability, and outstanding electrical, thermal and chemical resistance. It is transparent, flexible and elastic, and can be ground to very fine particles with a high aspect ratio²². The acrylic resin teeth produced for the removable dentures have a tendency to wear, which creates a problem. Therefore, we decided to investigate the wear of acrylic resin denture teeth by adding mica to denture teeth PMMA resin. Although used mica has an intermediate Mohs hardness, its broad particle size distribution and surface modification are believed to contribute a lot to abrasion resistance. Moreover, extra benefits are expected in easy taking of mould details and easy removal of mould material, as well as high solvent resistance due to the platelet nature of mica giving rise to lower viscosity and restricted diffusion of solvent molecules in layers²².

The present study was undertaken to determine the effect of mica as filler on *in vitro* 2-body wear of denture teeth PMMA resin. In addition, the effect of storage at open atmosphere on wear was studied.

Material and Method

Denture teeth acrylic resin (Rutinium Dental Manufacturing, Italy) was used in this study. The mica filler (10 µm in length, 32 µm in particle size) was supplied from DYO (The Yaşar Paint Group, Izmir, Turkey). The silane coupling agent, A-174, 3-methacryloxypropyl trimethoxy-silane (3-MPS) was supplied from Union Carbide, UK.

9 groups of specimens (totally 90 specimens) were divided as follows: 10 specimens of unfilled PMMA used as a control group (C), and the others were test groups of PMMA modified with 5, 10, 15, and 20% by weight unsilanized and silanized mica filler added to polymer (n=10 totally 80 specimens).

The specimen dimensions were 16 mm in diameter and 7 mm in thickness. Powder to liquid ratio recommended by the manufacturer was 20g/10ml. The powder to liquid ratio of the resin was 20 g of powder to 14 ml liquid, which was determined before the addition of mica fillers for all the groups. This higher than normal liquid to powder ratio was used to ensure better impregnation of the mica fillers. The mica fillers were set in pre-determined masses enough to form the percentages of 5, 10, 15, and 20% of the powder/liquid mixes. The weight of mica fillers was determined using an analytic balance to the nearest 0.0001 g (Precisa 3100C No 34709, Switzerland). The desired mass of fillers was first mixed thoroughly with a pre-determined weight of PMMA powder, then the required volume of methylmethacrylate liquid was added to the mixture and stirred so that the fillers oriented randomly to

give isotropic properties to the composite. The acrylic resin was polymerized at 175°C under a pressure of 160 bars for 3 minutes (Elimko 2200 Hydrocontrol Machine, Ankara, Turkey) and then cooled with water under a pressure of 160 bars for 3 minutes. After de-molding, the specimens were removed and finished to remove excess material by honing with fine emery paper. The exact final dimensions were ensured with a fine digital micrometer (Mitutoyo Digimatic Caliper 500154/CD 15 C, England).

The specimens were stored in distilled water at 37±1°C (Elektromag-M96K Water bath, Turkey) for 7 days before wear testing. The testing apparatus was an abrading device (APGI 613. 10 Carl Shroder KG Material prüfmachinen 6940 Weinheim Serial Number 40176) and was in compliance with DIN 53516 standard⁸. The Al₂O₃ emery paper was used as the abrading material (600 grid, 3M production resin paper 251 U-P 600). The emery paper was replaced before abrading each test group. All wearing tests were performed in distilled water at room temperature (23±1°C). The difference in weight measurements between before- and after-wear testing determined weight loss (Mettler H 20, Switzerland, nearest 0.01 mg). Weight loss (mg) was evaluated as amount of wear. The specimens were stored in a dry environment at room temperature for 90 days. After storage, the specimens were again stored in distilled water at 37±1°C for 7 days before repeating the wear test.

Mann Whitney U test, Wilcoxon test and Kruskal Wallis 1-Way Anova were conducted to analyze the findings. All analyses were executed using Unistat Statistical Package (5.1.03 Version, 1984-2001 Unistat Ltd, 4 Shirland Mews, London, England).

Results

Table 1 shows the means, standard deviations and statistical comparisons for the total weight loss (mg) of the control and test groups at initial and after-storage. Means were compared between control and test groups by Mann-Whitney U test. All the unsilanized test groups displayed a statistically significant higher total weight loss compared with the control group, both at the initial and after-storage at open atmosphere (p<0.05). On the contrary, compared to the control group, silanized test groups showed a statistically significant lower total weight loss (p<0.05).

Statistical comparison for total weight loss of the unsilanized and silanized test groups was performed by Wilcoxon test. Silanization significantly reduced (z=2.803; p=0.005) total weight loss both initially and after storage but 20% (z=2.803, p=0.07).

The effect of the filler concentrations on total weight loss (mg) of the test groups was compared by Kruskal Wallis 1-Way Anova (Tab. 2). No statistically significant difference in percentages was found among the silanized

mica incorporated groups both initially (p=0.07) and after storage (p=0.86). There was a statistically significant difference among unsilanized test groups (p=0.001). Total weight loss increased at a significant level between unsilanized 5% and 10% and also between unsilanized 15% and 20%; however decreased significantly between unsilanized 5% and 10% (p<0.05). After storage at open atmosphere, total weight loss again increased at a significant level between unsilanized 5% and 10% (p<0.05), whereas no difference was found among the other percentages (p >0.05).

Table 1. The means and standart deviations, and statistical comparison of the total weight loss (mg) of control and test groups

| | | Initial | After 90 days storage |
|-------------|-----|-----------------------|-------------------------|
| Control | C | 0.14 (0.02) | 0.14 (0.02) |
| Unsilanized | 5% | 0.15 (0.02) p: 0.023* | 0.16 (0.02) p: 0.008* |
| | 10% | 0.18 (0.03) p: 0.003* | 0.18 (0.02) p: 0.001* |
| | 5% | 0.16 (0.02) p: 0.016* | 0.17 (0.01) p: 0.001* |
| | 20% | 0.20 (0.03) p: 0.001* | 0.17 (0.05) p: 0.01* |
| Silanized | 5% | 0.10 (0.03) p: 0.005* | 0.11 (0.02) p: 0.002* |
| | 10% | 0.09 (0.03) p: 0.01* | 0.12 (0.03) p: 0.082 NS |
| | 5% | 0.12 (0.03) p: 0.028* | 0.12 (0.02) p: 0.016* |
| | 20% | 0.12 (0.03) p: 0.013* | 0.12 (0.03) p: 0.07 NS |

* - indicates significant difference between control and test groups at p<0.05 level

NS - indicates no significant difference at p>0.05 level

Table 2. Effect of filler quantity on the wear (mg) of the test specimens

| | Test | Unsilanized 5-20% | Silanized 5-20% |
|-----------------------|------------|-------------------|-----------------|
| Initial | Chi-Square | 17.036 | 6.809 |
| | Df | 3 | 3 |
| | Asymp. Sig | 0.001* | 0.078NS |
| After 90 days storage | Chi-Square | 8.060 | 0.757 |
| | Df | 3 | 3 |
| | Asymp. Sig | 0.045* | 0.860NS |

The statistical comparison of total weight loss of same test groups at initial and after 90 day storage was performed by Wilcoxon test (Tab. 3). Only the amount of wear of unsilanized 20% decreased at a statistically significant level after 90 day storage (p=0.02); the amount of wear of silanized 10% increased at a statistically significant level after storage (p=0.007). No significant difference was observed in the other groups (p>0.05).

Table 3. Statistical comparison of wear (mg) values of the same test groups initially and after storage

| | |
|-----------------|-----------------------|
| Unsilanized 5% | z:-0.968 p: 0.333(NS) |
| Unsilanized 10% | z:-0.255 p: 0.799(NS) |
| Unsilanized 15% | z:-1.886 p: 0.059(NS) |
| Unsilanized 20% | z:-2.191 p: 0.028* |
| Silanized 5% | z:-1.478 p: 0.139(NS) |
| Silanized 10% | z:-2.701 p: 0.007* |
| Silanized 15% | z:-0.051 p: 0.959(NS) |
| Silanized 20% | z:-0.255 p: 0.799(NS) |

Discussion

Assessment of wear resistance has been conducted through *in vivo* and *in vitro* methods^{5,7,15}. Due to complexity of the wear mechanism, correlation of the *in vivo* and *in vitro* finding is often difficult¹⁶. However, *in vitro* tests are still necessary to test newly developed materials, as the clinical trials are expensive and time consuming. *In vitro* methods are classified as a 2-body test^{3,13,19,23} or a 3-body test²⁵. The phenomena of occlusal wear under clinical conditions are generally divided into 2 components: an abrasive material loss found in contact free areas (CFA-wear 3-body wear) and a second type at the path of articulation on the occlusal contact areas (OCA-wear, 2-body wear)¹. The 2-body wear test was used in this study because wear resistance of the occlusal contact areas of removable prostheses was the subject of the study.

In our study it has been observed that the addition of untreated mica filler into similar matrix, PMMA, resulted in a detracton considerably from the wear resistance of unfilled rigid PMMA. Tappe et al^{29,30} stated that unsilanized and silanized quartz have a positive effect on the wear. Our results were not in accordance with their study. In our study, it was observed that unsilanized mica had a negative effect on wear, silanized mica had not. Our study disclosed a positive effect due to the addition of silanized mica filler only. It has been known that 3 factors appear to predominate in the effects of filler on wear resistance: the absolute hardness of filler, the strength of the interfacial bond between polymeric matrix and filler, and relative filler packing fraction. First factor is especially important in the cases where the absolute hardness of particles and abrasives are not comparable. Most abrasive media consists of silica corundum, which consists of hard, tough particles. If the filler is softer than the abrasive, it is readily abraded, whereas if it is harder than abrasive, the wear performance may be increased by inclusion of these particles into the matrix directly¹¹.

In this study, the decrease in wear resistance with mica loading may be attributed to the existence of the mica particles, which are softer than the Al_2O_3 abrasive in polymer matrix. It is then quite reasonable to expect further loss in wear resistance by an increase in mica loading for PMMA matrix. It is also clear from the results that silanization of mica surfaces overcome this drawback significantly *via* improving interfacial bonding, which makes the dislodgement of particles from the matrix difficult. Adequate adhesion of the fillers to the polymer matrix is one of the important variables for the strength of the composite².

When fillers are added to plastics, the bonding between them is very crucial. If the filler is silanized, the inter-bonding is stronger and more water-resistant, which in turn favours the mechanical properties of the material^{2,26,27}. The results of our study also support these findings. In our investigation, the specimens with silanized mica fillers displayed a significantly less wear compared with unsilanized specimens. In addition, the silanization of mica decreases the mica ratio in the composite *via* improving the wear resistance.

The incorporation of silanized mica higher than 5% did not decrease the wear significantly. Within these groups, the wear resistance increased up to 5% silanization and then almost level off. Moreover, since higher loading causes certain coloration, it seems to be useful to concentrate mainly around the composition loaded with 5% silanized mica. Similarly, quartz unsilanized and silanized average particle diameter of 15μ were added to artificial teeth made up of PMMA resin in different percentages of weight, and it was reported that as the proportion of quartz increased, the wear decreased. Addition of 15% quartz at maximum was recommended^{29,30}.

For a polymeric composite material, performance tests after aging are designed mainly in 2 different groups - the final performance of the sample after subjecting the material to: (a) specific conditions in which they are stored (shelf life), and (b) specific conditions that are simulated to environment in which it actively works. Possible degrading effect of both moisture and oxygen in open atmosphere on silane filler surface bonding *via* creating competitive hydrogen bonding sites, and on PMMA matrix *via* oxidative reactions, respectively, then resulting in loss of composite strength, was also investigated by repeating the wear tests with the samples waited at open atmosphere for a long time. Results given in table 3 show that the composites did not exhibit any difference in their wear strength, indicating a high moisture and oxygen resistance.

In general, measurements of the mica incorporated specimens after 90 days storage showed no difference in terms of wear. However, it is very interesting to note a minimum abrasion after 90 days in the group with 10% silanized mica, although initially remarkable increase in the wear was observed. In addition to this, the storage of the specimens that were reinforced with in a dry environment

for 90 days did not create a negative effect in general. The results of this study suggest that mica filler incorporated in denture teeth PMMA resin has relatively good stability against open atmosphere.

Conclusion

In conclusion, the silanized mica fillers in denture teeth PMMA resin enhanced wear resistance. The 5% silanized mica was found to give the best wear resistance result. There appeared to be a specific limit of mica fillers concentration above which the level of enhancement was reduced and resulted in undesirable coloration. In addition, unsilanized mica fillers actually reversed effect and weakened the resin.

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