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Effect of Mica and Glass on Acrylic Teeth Material's Color

Idil DIKBAS[1], Temel KOKSAL[1], Fatma UNALAN[2], Ozlem GURBUZ[3], Fuat NOYUN[1] and Ender KAZAZOGLU[1]
[1]Department of Prosthodontics, Faculty of Dentistry, Yeditepe University, Istanbul, Turkey
[2]Department of Prosthodontics, Faculty of Dentistry, Istanbul University, Istanbul, Turkey
[3]Department of Dentistry, Bakırkoy Mental Hospital, Istanbul, Turkey
Corresponding author, Idil Dikbas E-mail:idildikbas@yeditepe.edu.tr

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The purpose of this study was to evaluate the effect of two different ratios of silanized mica filler and milled glass fiber reinforcement on the color of acrylic denture teeth materials. Ten acrylic resin discs made of acrylic denture teeth material (PMMA) obtained from the manufacturer were used as the control group. Four experimental groups were modified from the control group's PMMA material by adding a ratio of 5% or 10% by weight of silane-treated mica filler or milled glass fibers. Each group consisted of 10 specimens. Measurements were performed using a spectrophotometer CM-2600d, and the color changes were characterized in the Commission Internationale d'Eclairage L*a*b* color space. ΔE^* values of 5% mica-, 10% mica-, 5% glass-, and 10% glass-containing sample groups were 2.46, 3.03, 2.16, and 2.59 respectively. There were statistically significant differences in L*, a*, and b* values between the control group and each test group. It was shown that when PMMA denture teeth material was modified with silane-treated mica filler or silane-treated milled glass fibers for the purpose of reinforcement, it would also cause significant changes to the original color of the material.

Key words: Color, Spectrophotometer, Acrylic denture teeth

INTRODUCTION

Acrylic resin teeth, which have been widely used in prosthetic dentistry, have some advantages over porcelain teeth. Acrylic denture teeth made of polymethyl methacrylate (PMMA) have excellent fracture toughness, allow easy occlusal adjustment, and demonstrate high bond strength with denture base materials. However, their wear resistance poses a problem[1−3]. To overcome this problem, different types of material have been added to polymer materials to improve their mechanical properties and wear resistance[1,4] − namely carbon fibers[5], aramid fibers[6], glass fibers[7−9], and mica fillers[10,11].

Inorganic fillers in PMMA are subjected to silane coupling treatment to enhance the adhesion of these particles to the matrix base of the teeth material[12−15]. Unalan *et al.*[11] found that addition of silanized mica in the ratios of 5% and 10% significantly decreased the wear rates of PMMA. Likewise, Gurbuz *et al.*[9] observed that addition of similar ratios of glass fibers caused the wear rates of PMMA to decrease significantly. However, reinforcement additives might change the original color of denture teeth material (PMMA). The purpose of this study, therefore, was to evaluate the effect of two different ratios of silanized mica filler and milled glass fibers on the color of acrylic denture teeth materials.

MATERIALS AND METHODS

Test materials

Denture teeth material, PMMA (Ruthinium Dental Manufacturing, Italy), was supplied in the lightest shade in both powder and liquid forms. Acrylic resin discs made of PMMA were prepared according to the manufacturer's instructions. These specimens were used as the control group. Four experimental groups were modified from the control group's PMMA material by adding two different ratios (5%, 10%) of silane-treated mica filler and two different ratios (5%, 10%) of silane-treated milled glass fibers by weight. As a result, one control and four experimental groups were obtained (Table 1). Each group consisted of 10 samples. The dimensions of each sample were 16 mm in diameter and 7 mm in thickness.

For the silane-treated muscovite mica filler (DYO Boya Fabrikalari Sanayii ve Ticaret A.Ş., İzmir, Turkey) used in this study, its physical prop-

Table 1 Contents of control group and test groups

Group	Content
Control group	PMMA
Test group 1	5% mica filler-added PMMA
Test group 2	10% mica filler-added PMMA
Test group 3	5% glass fiber-added PMMA
Test group 4	10% glass fiber-added PMMA

PMMA: Polymethyl methacrylate

erties were: *ca.* 32 μm in particle size, 2.7 g/cm^3 in density, and 2.5 in Mohs hardness. As for the silane-treated milled E glass fibers (Cam Elyaf A.Ş., Çayırova, Turkey), the physical properties were: 32 μm in particle size, 1.2 μm in diameter, 0.8 mm in length, 2.54 g/cm^3 in density, and 6.5 in Mohs hardness. The silane coupling agent, A-174 (which contained 3-methacryloxypropyl trimethoxy silane, 3-MPS), was supplied from Union Carbide, UK.

Specimen preparation

Control group specimens were prepared in 20 g/10 ml powder/liquid (P/L) ratio according to the manufacturer's recommendations. To incorporate mica into PMMA material, the P/L ratio was decreased to 20 g/14 ml. This modified P/L ratio was used to ensure better impregnation of the mica filler. For the impregnation of glass fibers, it was not necessary to change the control group's P/L ratio. To obtain mixtures containing 5% and 10% ratios of silane-treated mica filler and silane-treated glass fibers by weight, required weights of these additives were predetermined.

Initially, the control group was prepared according to the predetermined P/L ratio. Then, the required additive amount of each test group was added to the predetermined weight of PMMA powder and mixed thoroughly. Following which, the required volume of PMMA liquid was added to the mixture and stirred so that the additives were randomly oriented to give isotropic properties to the composite. The flasked acrylic resin dough was polymerized at 175 ℃ under a pressure of 160 bar for three minutes (Elimko 2200 Hidrocontrol Machine, Ankara, Turkey) and then cooled with water under a pressure of 160 bar for three minutes. After demoulding, specimen surfaces which were to be used for color measurements had a glossy texture. These specimens were stored in distilled water at 37±1℃ in a closed, dark box until evaluation.

Color change measurement

After removal from the distilled water, the color of each group's specimens was measured by a spectrophotometer, CM 2600-d (Konica Minolta Sensing, Inc., Japan).

Before measurement, the colorimeter was calibrated according to the manufacturer's instructions by using the supplied white calibration standard. Measurements were taken at three different points of each sample. The mean value of these three measurements was automatically calculated by the spectrophotometer and recorded.

Color changes were characterized in the Commission Internationale d'Eclairage L*a*b* color space (CIE L*a*b*). The CIE L*a*b* system was employed in this study because it is well suited for the determination of small color differences[16]. Direction of a color difference is described by the magnitude and algebraic sign of each component − ΔL^*, Δa^*, and Δb^* − as follows:

$$\Delta L^* = L_{test} - L_{control},\ \Delta a^* = a_{test} - a_{control},\ \text{and}\ \Delta b^* = b_{test} - b_{control}$$

where $L_{control}$, $a_{control}$, and $b_{control}$ refer to the color values of the control samples, while L_{test}, a_{test}, and b_{test} refer to the color values of the test samples. All the samples of the four experimental groups were randomly matched to the control group's specimens, and each test group's samples had their respective ΔL^*, Δa^*, and Δb^* values.

For each of these components − ΔL^*, Δa^*, and Δb^*, their signs bear the following approximate meanings: $+\Delta L^*$=lighter, $-\Delta L^*$=darker, $+\Delta a$=redder (less green), $-\Delta a$=greener (less red), $+\Delta b$=yellow (less blue), and $-\Delta b$=bluer (less yellow)[17]. As for the color difference between the control and test samples, it was expressed as a single parameter, ΔE^*, which was calculated from the following formula[5,18]:

$$\Delta E^* = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

To relate the amount of color change (ΔE^*) recorded by the spectrophotometer to a clinical environment, the data were converted to National Bureau of Standards units (NBS units) through the following equation:

$$\text{NBS units} = \Delta E^* \times 0.92$$

where critical remarks of color differences as expressed by NBS units are shown in Table 2[19].

Statistical analysis

Color change between control and each test group was statistically analyzed using Mann-Whitney U test. Mean ΔL^*, Δa^*, and Δb^* values of test groups were compared by Kruskall-Wallis test at the 95% confidence level.

RESULTS

Color values of the control and test groups are shown in Table 3. Table 4 shows the differences between the control and each test group in L*, a*, and b* values (*i.e.*, ΔL^*, Δa^*, and Δb^* respectively), as well as the ΔE^* value of each test group. As shown

Table 2 National Bureau Standards (NBS) ratings

NBS unit	Critical remarks of color differences	
0.0−0.5	Trace:	Extremely slight change
0.5−1.5	Slight:	Slight change
1.5−3.0	Noticeable:	Perceivable change
3.0−6.0	Appreciable:	Marked change
6.0−12.0	Much:	Extremely marked change
12.0 or more	Very much:	Change to other color

in Table 4, there were no significant differences among the test groups' values ($p>0.05$).

Table 5 shows the color difference value (ΔE^*) and its corresponding NBS value for all test groups. According to this table, the NBS ratings of all test groups were within the range of 1.5-3.0, which meant that a noticeable color change was observed. In other words, test samples containing 5% mica, 10% mica, 5% glass, and 10% glass showed noticeable color differences when compared to the control group samples.

Table 6 shows the dual comparison results between each test group and control group. In each test group, L^*, a^*, and b^* values were significantly different from the control group's corresponding values ($p<0.05$).

Table 3 Color values of the control and four test groups

		L^*	a^*	b^*
Control	Mean	69.27	0.45	11.76
	SD	0.73	0.19	0.41
Test group 1 (5% mica)	Mean	67.12	0.99	12.53
	SD	0.51	0.11	0.56
Test group 2 (10% mica)	Mean	66.64	1.19	12.80
	SD	0.98	0.34	0.30
Test group 3 (5% glass)	Mean	67.29	0.88	12.24
	SD	0.64	0.17	0.39
Test group 4 (10% glass)	Mean	67.10	0.96	12.90
	SD	0.55	0.14	0.29

SD: Standard deviation

Table 4 Differences between control and each test group in L^*, a^*, and b^* values, as well as the ΔE^* value of each test group and Kruskall-Wallis results

		Group 1 (5% mica)	Group 2 (10% mica)	Group 3 (5% glass)	Group 4 (10% glass)	KW	P
ΔL^*	Mean	−2.15	−2.62	−1.98	−2.17	1.51	0.678
	SD	0.72	1.16	0.68	0.79		
Δa^*	Mean	0.54	0.73	0.42	0.51	4.55	0.216
	SD	0.22	0.40	0.27	0.21		
Δb^*	Mean	0.77	1.04	0.48	1.14	7.73	0.052
	SD	0.62	0.48	0.48	0.48		
ΔE^*	Mean	2.46	3.03	2.16	2.59	5.20	0.157
	SD	0.56	0.98	0.62	0.66		

P>0.05: Insignificant
SD: Standard deviation
KW: Kruskall-Wallis statistical test

Table 5 Color changes of test groups

	ΔE^*	NBS unit	Remark of color difference
Test group 1 (5% mica)	2.46	2.26	Noticeable
Test group 2 (10% mica)	3.03	2.78	Noticeable
Test group 3 (5% glass)	2.16	1.99	Noticeable
Test group 4 (10% glass)	2.59	2.38	Noticeable

NBS: National Bureau of Standards

Table 6 Dual comparison of each test group to control group

	L* Mean (SD)	a* Mean (SD)	b* Mean (SD)
Control	69.27 (0.73)	0.45 (0.19)	11.76 (0.41)
5% mica	67.12 (0.51)	0.99 (0.11)	12.53 (0.56)
MW	0	0	13
P	0.0001	0.0001	0.005
Control	69.27 (0.73)	0.45 (0.19)	11.76 (0.41)
10% mica	66.64 (0.98)	1.19 (0.34)	12.80 (0.30)
MW	0	0	1
P	0.0001	0.0001	0.0001
Control	69.27 (0.73)	0.45 (0.19)	11.76 (0.41)
5% glass	67.29 (0.64)	0.88 (0.17)	12.24 (0.39)
MW	3	4	20
P	0.0001	0.001	0.023
Control	69.27 (0.73)	0.45 (0.19)	11.76 (0.41)
10% glass	67.10 (0.55)	0.96 (0.14)	12.90 (0.29)
MW	0	1	1
P	0.0001	0.0001	0.0001

SD: Standard deviation
MW: Mann-Whitney U statistical test

DISCUSSION

Color changes can be evaluated visually as well as by instrumental techniques. Since instrumental measurements eliminate the subjective errors in visual color comparison, spectrophotometers were used instead of the Munsell color order system for visual evaluation[20].

The CIE* L*a*b* color order system provides a useful tool for quantifying the color properties of dental materials. Color is described using a mathematical three-dimensional system based on an equal distance in the color space that is directly correlated with equal perceived gradations. This system divides color into three attributes. L* is a measure of whiteness or brightness. a* measures the hue-chroma in the red-green direction, while b* measures hue-chroma in the yellow-blue axis. High L* values are obtained for bright or white samples. Positive a* values are red, negative values are green. Positive b* values are yellow, negative values are blue[21].

Color difference (ΔE^*) was calculated from the formula previously mentioned. However, there are various approaches to assessing the ΔE^* value. It has been reported by Seghi *et al.*[22] that a ΔE^* value equal to 1 was considered visually detectable in 50% of the cases, whereas a ΔE^* value greater than 2 was detectable in all cases. Um and Ruyter[20] also suggested that a ΔE^* value equal to 1 was "visually perceptible". Similarly, Liberman *et al.*[23] concluded that a value of $\Delta E^*=1$ should be sufficient to discriminate between color stability and perceptible change.

On the other hand, in a study performed by Yannikakis *et al.*[24], a ΔE^* value ≤ 2 was considered "visually imperceptible", whereas a ΔE^* value greater than 2 was used as a baseline and stated as "visually perceptible". Similarly, Stober *et al.* claimed that only color differences with ΔE^* ranging from 2 to 3 were visible[25]. On the same note, Ikeda *et al.*[26] stated that ΔE^* values between 0 and 2 were imperceptible, values between 2 and 3 were just perceptible, values between 3 and 8 were moderately perceptible, and values above 8 were markedly perceptible. A color change that is more than perceptible (*i.e.*, $\Delta E^*>1.0$)[20] will be considered as acceptable up to the value of $\Delta E^*=3.3$, which is considered to be the upper limit of acceptability in subjective visual evaluations[27]. Indeed, Ruyter *et al.*[27] described discolorations of $\Delta E^*>3.3$ as no longer clinically acceptable. In a study by Yannikakis *et al.*[24], it was shown that for two similar looking − but different − samples based on a porcelain shade guide, the color difference was found to be 1.85 units.

Although ΔE^* values are accepted as measures of color change, it is difficult to equate these numeric values and the accompanying degree of color change to a clinical setting and the perception of color change by an observer. One method frequently used to determine the degree of color difference and thus a practical application of ΔE^* color notation is to convert it to National Bureau of Standards (NBS) units. NBS critical remarks of color differences are used for color comparison and quality control func-

tions, because only the ΔE^* value needs specification rather than a range of L^*, a^*, and b^* values. However, with these values, it is possible to determine color changes that occur in test materials[19].

In the present study, the authors preferred to determine the differences in L^*, a^*, and b^* values between the control group and each test group. Following which, ΔE^* values which showed substantial color differences were also calculated. In addition, corresponding NBS units were calculated to assess clinical acceptability.

As seen in Tables 3 and 4, changes in L^* value of all test groups were toward the negative direction, which meant that addition of both mica and glass resulted in loss of brightness. When the mean L^* value of the control group was compared to the mean L^* value of each test group, maximum difference was observed with the 10% mica-containing group ($\Delta L^* = -2.62 \pm 1.16$) while minimum difference was seen in the 5% glass-containing group ($\Delta L^* = 1.98 \pm 0.68$). Similar results were obtained with the Δa^* value. The 5% glass-containing group yielded the lowest difference ($\Delta a^* = 0.42 \pm 0.27$) while the 10% mica-containing group yielded the highest difference ($\Delta a^* = 0.73 \pm 0.40$). On the overall, the a^* values of all test groups changed towards redness. In the b^* chromaticity coordinate, the highest change was in the 10% glass-containing group ($\Delta b^* = 0.14 \pm 0.48$). It meant that this group showed a change that was yellower. In summary, there were statistically significant differences in the L^*, a^*, and b^* values between the control group and each test group.

ΔE^* values of 5% mica-, 10% mica-, 5% glass-, and 10% glass-containing groups were 2.46, 3.03, 2.16, and 2.59 respectively. Some authors[20,28,29] assumed that values of $\Delta E^* < 1$ were regarded as not appreciable by the human eye. Values of $1 < \Delta E^* < 3.3$ were regarded as appreciable by skilled operators, but considered clinically acceptable. However, values of $\Delta E^* > 3.3$ were considered appreciable even by non-skilled persons, and for that reason considered as clinically unacceptable[20,28,29]. Therefore, ΔE^* values found in this study were supposed to be clinically acceptable. At this juncture, it must be highlighted that this result is valid for discolorations originating from staining agents. But for reinforcement materials added to denture teeth material (such as PMMA), it is desirable that the original color of PMMA be preserved. In this study, although the color differences were clinically acceptable, the L^* values of all test groups changed towards the darker side − which meant that the lightest shade of PMMA was lost.

When the results were evaluated in terms of NBS units of color difference, the 10% mica-containing test group exhibited the greatest chromatic change (2.78). The lowest chromatic change belonged to the 5% glass-containing test group (1.99) (Table 5). Nonetheless, according to NBS ratings (Table 2), the color difference of all test groups was considered noticeable (*i.e.*, perceivable change). In other words, this clinical remark also indicated that the original shade of PMMA had changed. Therefore, it should be noted that if denture teeth were reinforced by adding mica filler or glass fibers, then even the denture teeth material of the lightest shade on the shade scale will yield a resultant color that is darker than its original shade.

CONCLUSIONS

The effects of two reinforcement additives in two different ratios were evaluated from the perspective of the color stability of the original PMMA denture teeth material. Within the limitations of this *in vitro* study, the following conclusions were drawn:

1) When PMMA denture teeth material was modified by silane-treated mica filler or silane-treated milled glass fibers for the purpose of reinforcement, significant changes to the original color of the material occurred, as shown in the L^*, a^*, and b^* values.

2) All test groups exhibited "noticeable" color changes according to NBS critical remarks of color differences.

3) 10% mica-containing test group exhibited a greater chromatic change than the other test groups.

4) Denture teeth manufacturers are therefore encouraged to supply reinforcement materials which do not affect the selected original shade of PMMA.

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