COLOR PROPERTIES OF POLYMETHYLMETHACRYLATE MATERIAL INCORPORATED WITH GOLD NANOPARTICLES

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Abstract: The aim of this study was to investigate the influence of the addition of different concentrations of gold nanoparticles (AuNPs) on color parameters of nano-modified polymethyl methacrylate (PMMA) prosthesis base material as well as their mutual comparison. In our investigation, we used AuNPs produced by new technologies Ultrasonic Spray Pyrolysis (USP) method, from the precursor solution from Au (III)acetate. The addition of highly dispersed AuNP in PMMA (ProBase Hot, Ivoclar Vivadent, Liechtenstein), followed by the formation of nano-modified polymer composite is performed in a special way. Four groups of samples by uniform dimensions of 50 x 50 x 4 mm, and with different AuNPs concentration (PMMA-AuNP1 0,12%, PMMA-AuNP2 0,43%, PMMA-AuNP3 0,74% and control group) were performed. The color values show that L * (light) PMMA-AuNP1 sample is slightly lower than the base sample, while the PMMA-AuNP2 and PMMA-AuNP3 have a higher value. The basic sample has the highest C * value, so it is the most saturated (most colorful). The basic sample also has the highest value of b * (more yellow shades), while the other three samples have a lower b * value (more blue shades). Also, the color angle h is smaller than the basic one for all three samples (closer to the red color axis a +). Color changes in base resins were significantly affected by the addition of gold nanoparticles (p>0.05). The color differences ΔE * are in the range from 2,6 – 4,9. The addition of AuNPs at the tested concentrations does not have a statistically significant effect on the change in material translucency of PMMA/AuNPs nanocomposite.

Keywords: gold nanoparticles, PMMA, denture acrylic resin, color properties.

1. INTRODUCTION

Prosthodontics is a branch of dentistry assisted with the growing knowledge and technologies to obtain the requirement of perfect aesthetics in patients. Dental aesthetics plays a very important role as their demand steadily grows to restore the dentition naturally [1].

One of the most commonly used dental materials in prosthodontics is poly(methyl methacrylate) (PMMA). It is the material of choice for the denture base because of its good properties, such as simplicity of fabrication technology, reparation capabilities, good polishing capabilities, stability in the oral environment, biocompatibility, satisfactory aesthetics and affordable prices [2–5].

Recent studies have investigated the effect of incorporating inorganic nanoparticles into PMMA to improve its properties. The shape and size, as well as the concentration and interaction of these nanoparticles with a polymer matrix, determine the properties of a polymer nanocomposite mixture [1,5–7]. Ideally, the material used to enhance the properties of PMMA improve the mechanical properties without causing an adverse reaction to the aesthetics [8].

Nanocomposites are described as two-phase materials where one of the phases has at least one dimension in the nanometer range (1–100 nm) [9]. Nanomaterials are known for their superior performance over conventional materials. When nanoparticles are incorporated into a polymeric matrix as fillers, the characteristics of both ingredients are integrated to improve the optical and mechanical properties of the nanocomposites [6].

Color is one of the most important attributes of aesthetic restorations. Matrix, filler composition, filler content, minor pigment addition, initiation components, and filler coupling agents affect the color of aesthetic materials. The interactions of each of these components may have a role in the color stability of the material (10).

Good optical properties of PMMA reinforced with inorganic fillers are vital. PMMA permits filler reinforcements, which may influence the translucency properties and, consequently, the aesthetic values [6].

Translucency is the property of a substance that permits the passage of light but disperses the light so that objects cannot be seen through the material; therefore, it could be described as partial opacity or a state between complete opacity and complete transparency [11].

The translucency property for any material results from the color difference between the thickness of the test material on a white background and the equal thickness of that material over a black background. This color difference provides some value concerning the shared visual perception of translucency [12].

Color changes can be evaluated by visual or instrumental techniques. However, color evaluation by visual comparison may not be a reliable method due to inconsistencies inherent to color perception and specification between observers. Instrumental techniques for color measurement include colorimetry, spectrophotometry and digital image analysis, where spectrophotometry is the most reliable technique in dental material studies [10].

The CIE L * a * b * color system will be used in this study to evaluate translucency and color difference between samples. All colors in nature are obtained through a blending of three basic colors i.e., red, green, and blue in certain proportions. The CIE L*a*b* system has been developed based on this practice [13]. This study aimed to investigate the effect of AuNPs addition on the color and translucency of the PMMA/AuNPs denture base material.

2. MATERIALS AND METHODS

2.1. Sample preparation

In our study, we used AuNPs produced by the modern method of ultrasonic spray pyrolysis (USP). Gold (III) acetate (Au (CH ₃ COO)₃ was used as a precursor solution. Polyvinylpyrrolidone (PVP) was used as a stabilizer for AuNPs. In the previous work, Rudolf et al. used the same method of synthesis of AuNPs by USP pyrolysis [14].

In this study, to suppress the formation of agglomeration, the prepared concentrated AuNPs solution was dispersed in the acrylic liquid methylmethacrylate (MMA) in the desired ratio and then mixed with the powder part (PMMA) of the acrylic material.

A conventional heat-polymerized PMMA (ProBase Hot, Ivoclar Vivadent, Liechtenstein) (Figure 1) was used as a matrix component and the AuNPs (Figure 2) as reinforcing agents. Based on similar studies, 24 samples were prepared for the present *in vitro* study. The specimens were categorized into four groups (n=6). Group PMMA was the control group (unmodified heat-polymerized PMMA). The specimens of the remaining three groups (PMMA-AuNPs 0. 12%, PMMA-AuNPs 0. 43%, PMMA-AuNPs 0. 74%) were modified with the addition of different concentrations AuNPs.

The synthesized AuNPs were added to the MMA at 0.12, 0.43 and 0.74 wt% and sonicated for 15 min. Then, the solution (MMA with AuNPs) was mixed with the powder portion of PMMA according to the manufacturer's recommended polymer and monomer ratio and they were mixed by hand using a stainless-steel spatula to make sure all the powder was uniformly distributed within the resin monomer.

The resin was left in the closed mixing jar until it reached the dough stage, and then, the mixture was kneaded thoroughly to a homogeneous dough. The dough was packed into the metal molds to create square-shaped ($50 \times 50 \times 3.0 \pm 0.5$ mm) specimens for color testing and circular-shaped ($15 \times 2.0 \pm 0.5$ mm) ones for translucency testing. The ask metal lid was closed properly and placed under 80 bars of pressure in a bench press to compress the material into the mold. Polymerization was carried out by heat in a water bath at 100 °C for 45 minutes. After completion of the polymerization cycle, the metal molds are allowed to cool in the same water bath to room temperature. After cooling, the specimens were removed from the metal molds. After



Figure 1. Heat-polymerized PMMA ProBase Hot, IvoclarVivadent, Liechtenstein

2.2. Color measurements

A spectrophotometer (UV-VIS SF 600 Plus, Datacolor, Switzerland) was used to measure the color changes with CIE L * a * b * color system. This system is based on three color-defining parameters: L *, a * and b *, L * represents brightness, a * represents red-green and b * represents yellowblue. ΔE present a value for color differences. ΔE was calculated from the values of L *, a * and b * obtained on black background between the control and experimental groups using the following equation (1):

$$\Delta E = [(L1^* - L2^*)2 + (a1^* - a2^*)2 + (b1^* - b2^*)2]1/2$$
(1)

(L1, a1, b1: samples with AuNPs, L0, a0, b0: control samples) A high ΔE value indicates a large color difference. Three measurements were made on each sample and the average value was recorded.

removing molds and trimming the edges, the specimens were polished with 800-, 1000-and 1200- grit abrasive paper to obtain polished surfaces.



Figure 2. AuNPs

2.3. Translucency test

Reflection values were measured using a spectrophotometer (UV-VIS SF 600 Plus, Datacolor, Switzerland). Each sample was fixed on a black and white background before measurement. A CIE L * a * b * color system was used to evaluate changes in translucency. Three readings were made for each sample and the average was automatically presented by the software. The data are tabulated and the translucency is calculated using the following equation (2):

TR = [(L * white-L * black) 2+ (a * white *)black) 2+ (b * white-b * black) 2] 1/2(2) where TR is translucency.

2.4. Statistical analysis

The color and translucency data were statistically analyzed by using a one-way ANOVA test and paired t-tests. For all statistical tests, a result was considered statistically significant at p > 0.05.

3. RESULTS

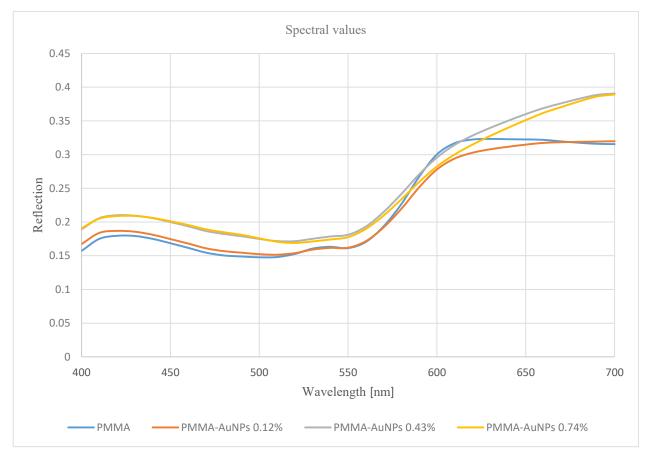
The results show that the PMMA-AuNP1 sample is slightly darker than the base sample, while the PMMA-AuNP2 and PMMA-AuNP3 samples are brighter, and all three samples are less saturated than the base sample (lower C * value). Also, the color angle h is smaller than the basic

one for all three samples (closer to the red color axis a +)

The color differences ΔE^* are in the range from 2.6 to 4.9, (Table 3) which is also noticeable with the bare eye. We can conclude that the PMMA-AuNP2 and PMMA-AuNP3 samples are most consistent with the human gum color.

Table 1. The color differences (ΔE) of nanocomposite PMMA/AuNPs specimens

GROUPS	L*	A*	B*	C*	Н
РММА	51.76673	17.61275	6.89255	18.9134	21.3723
PMMA-AUNPS 0. 12%	51.34773	16.14761	4.79046	16.8432	16.5239
PMMA-AUNPS 0. 43%	53.7196	16.13301	3.79899	16.5743	13.2506
PMMA-AUNPS 0. 74%	53.1638	15.48815	2.65671	15.7144	9.73332



Graph 1. Color spectral values

Table 2. Color differences $\Delta E *$ between samples

GROUPS	MEAN VALUES \pm SD	P-VALUE
РММА	-	-
PMMA-AUNPS 0.12%	2.596 ± 0.11	0.012
PMMA-AUNPS 0.43%	3.946 ± 0.29	0.310*
PMMA-AUNPS 0.74%	4.940 ± 0.31	0.573*

: Clinically acceptable value under the criterion of ΔE^ (2. 69) * Significance p>0.05

SD= standard deviation

Table 3. Translucency values (TR)

GROUPS	MEAN VALUES ± SD	P-VALUE
PMMA	12.80 ± 0.59	-
PMMA-AUNPS 0.12%	12.00 ± 0.48	0.013
PMMA-AUNPS 0.43%	11.65 ± 0. 45	0.021
PMMA-AUNPS 0.74%	11.12 ± 0. 40	0.044

*Significance p>0.05

SD= standard deviation

4. DISCUSSION

A spectrophotometer is one of the methods that can quantitatively assess material color changes, provide an objective evaluation, and express color objectively. It was used in the evaluation of color stability and translucency and was certified by the International Commission on Illumination in 1978 [15].

The color difference ΔE^* of tested samples ranged from 2.59 \pm 0.69 to 4.94 \pm 1.38 compared with control. The results show that no statistically significant change in color was observed in the first experimental group (PMMA-AuNPs 0.12%) according to the ΔE^* criteria (Table 2). The second and third experimental groups (PMMA-AuNPs 0.43% and PMMA-AuNPs 0.74%) showed a statistically significant change in color according to the ΔE^* criteria (Table 2). The color change is in direct proportion to the concentrations of AuNPs in PMMA (Table 2).

The optical properties of dental materials are of particular importance as these materials must meet high aesthetic criteria [16]. The heat-cured acrylic resin comes in a translucent pink color, which is then mixed with a monomer and undergoes heat polymerization. In general, the PMMA is translucent, it has good biocompatibility and low toxicity. Despite its ease of manipulation and low cost, it is prone to several disadvantages, such as poor mechanical and surface properties, resulting in denture fracture, porosities, plaque accumulations, and stains. To overcome these weak properties, PMMAis reinforced with AuNPs to improve the mechanical properties without adversely affecting the aesthetics [6]. Color represents the optical property of dental restorative materials, that is, the quality of an object to reflect and conduct light and is considered a significant parameter for the aesthetic appearance of dentures since color change acts as an indicator of material ageing or damage [17].

Color perception is a psychophysical phenomenon with variations, both between individuals and within an individual at different times. The CIELAB color system is used almost exclusively for color research in dentistry around the world. The strength of this system, unlike that of the Munsell system, is its capability for clinical interpretation, at equal distances across the CIELAB color space (color differences or ΔE) represent approximately uniform steps in human color perception, improving the interpretation of color measurements. Thus, the application of this system assisted the study to have a precise method of color evaluation [18].

Color variations are investigated through the change in color parameters L* (lightness-darkness), a* (red-green), and b* (yellow-blue) of the CIELAB system, where the mean value ΔE ab corresponds to the color difference and gains an arithmetic value. Great controversy exists regarding the exact ΔEab value that is clinically acceptable and not perceivable to either patients or dentists. It has been reported that some individuals perceive color differences as low as 0.5, whereas others do not see differences of ΔEab equal to 4.19 According to Douglas et al. 20 $\Delta Eab < 2$ means color matching, while a threshold value of $\Delta Eab >$ 3.3 is considered to be a clinically distinguishable color difference in the intraoral environment [19]. According to the National Bureau of Standards, a color change is considered to be very low when ΔE is less than 1, clinically acceptable when ΔE is between 1 and 3, and clinically noticeable when ΔE exceeds 3 [20].

Chang et al also consider the clinically acceptable ΔE * value to be below 2.69, all over this are considered clinically unacceptable [21].

Kuehni and Marcus and Seghi *et al*.concluded from their research that an ΔE * value equal to 1 is considered visually detected 50% of the time, while an ΔE * value greater than 2 can be detected 100% of the time [22,23]. Um and Ruiter also noted that the ΔE * value of 1 is "visually observable" [24].

Gross and Moser reported that it was impossible to know when the ΔE value was between 0 and 2. Ruyter and Buyukyilmaz suggested that acceptable ΔE values were less than 3.3. Eldiwany et al. showed that the ΔE value of 3.3 or more is visually perceptible. Goldstein and Schmitt concluded that if the ΔE value is greater than 3.7, the clinical tolerance limit is exceeded and the difference is visible [15]. The results of this research were presented in accordance with the recommendations of the National Bureau of Standards [20]. The first experimental group had a clinically acceptable value $\Delta E * = 2.59$, while the other two groups had a higher value (the second group $\Delta E * = 3.94$, the third group $\Delta E * = 4.94$), i.e., there was a clinically noticeable discoloration.

In his research, Nam Ki obtained similar results; he concluded that the color of the samples changed depending on the concentrations of AuNPs and AgNPs in the samples, that is, as the concentration of AuNPs and AgNPs increased, the color difference compared with the control sample increased. The largest color change was obtained with the highest concentration of AuNPs in the samples (200 ppm) with $\Delta E = 22.56$ [25]. The results of the present study agreed with those of previous ones De Matteis V et al. De Matteis V et al. added 3 wt% and 3.5 wt% AgNPs to heatpolymerized acrylic resin, and they observed color difference compared with the control group. The most noticeable color difference was observed in the samples with the highest concentration of silver nanoparticles in PMMA [26].

It is well known that discoloration is a common problem for gold and silver-containing materials; therefore, further studies are required to investigate the improvement of color stability for the clinical application possibility so as not to compromise the aesthetics of the prosthesis [27].

The translucency parameter (TP) is one of the parameters used to characterize the optical properties of esthetic materials [28].

Denture base materials should have a color similar to normal soft tissues, but also a translucency that will allow the light to pass through and reflect normal tissue shades for a more natural appearance. Both color and translucency of denture base materials are maintained throughout their clinical use [17].

A higher value for the translucency parameter represents higher translucency; with increasing opacity, the value of the translucency parameter decreases, that is, when the measured translucency approaches zero it is considered that the material is completely opaque [10,11]. The results of this study show that the addition of AuNPs to PMMA does not lead to a statistically significant decrease in the translucency of the experimental groups (PMMA-AuNPs 0.12%, PMMA-AuNPs 0.43% and PMMA-AuNPs 0.74%) compared to the control group (Table 3). As part of their research, Gad *et al.* examined the translucency of zirconium oxide nanocomposites. They concluded that zirconium oxide nanoparticles in PMMA decrease the translucency of nanocomposites and that the decrease in translucency is proportional to the increase in the concentration of zirconium oxide nanoparticles in PMMA [6].

No studies in the literature have examined the translucency of PMMA/AuNPs nanocomposites. Therefore, it is not possible to compare the results of the current study and previous similar studies.

5. CONCLUSION

In accordance with the limitations of this study, the following conclusions can be drawn:

The addition of AuNPs to PMMA influences the color change of the base material. The color differences are directly proportional to the concentrations of AuNPs in PMMA. Therefore, caution should be exercised in selecting the appropriate concentration of AuNPs for nano-modification of PMMA to fabricate the denture base so as not to adversely affect the aesthetic parameters. The addition of AuNPs at the tested concentrations does not have a statistically significant effect on the change in material translucency of PMMA/AuNPs nanocomposite.

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КАРАКТЕРИСТИКЕ БОЈЕ ПОЛИМЕТИЛМЕТАКРИЛАТА ИНКОРПОРИРАНОГ СА НАНОЧЕСТИЦАМА ЗЛАТА

Сажетак: Циљ овог рада био је да се испита утицај додавања различитих концентрација наночестица злата (AuNPs) на параметре боје наномодификованог основног материјала полиметилметакрилата (PMMA) протезе као и њихово међусобно поређење. Користили смо AuNPs произведене новим технологијама - методом ултразвучне спреј пиролизе (УСП), из раствора прекурсора Ау (III) ацетата. Додавањем високо диспергованих AuNPs, у PMMA (ProBase Hot, Ivoclar Vivadent, Liethenstein) извршено је формирање нанокомпозита. Експерименталне узорке подијелили смо у четири групе. Три експерименталне групе са различитим концентрацијама AuNPs (I група – 0,12 wr.%, II група – 0,43 wr.%, III група – 0,74 wr.%) и једна контролна група. Укупно је направљено 24 узорка и распоређено у сваку групу по шест узорака (н = 6). Додавањем високо диспергованих AuNPs у PMMA добили смо наномодификовани полимерни композит. Резултат показује да је вриједност L * (light) узорка PMMA-AuNP1 нешто нижа од контролног узорка, док РММА-AuNP2 и РММА-AuNP3 имају веће вриједности. Контролни узорак има највишу С * вриједност, тако да је најзасићенији (најсвјетлији). Контролни узорак такође има највећу вриједност б * (више жутих нијанси), док остала три узорка имају нижу b * вриједност (више плавих нијанси). Такође, угао боје h је мањи од контролног узорка за сва три експериментална узорка (ближе оси црвене боје а +). На промјену боје у базним смолама значајно је утицало додавање наночестица злата (p >0,05). Разлике у боји ∆Е * су у опсегу 2,6–4,9. Додавање AuNP-а у тестираним концентрацијама нема статистички значајан утјецај на промјену транслуценције нанокомпозита PMMA/AuNPs.

Кључне ријечи: наночестице злата, РММА, протеза акрилна смола, особине боје.

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