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Investigation of the Thermal Conductivity and Flexural Strength of Polymethylmethacrylate Denture Base Material with SiC and Al_2O_3 Added

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Abstract. *Although polymethylmethacrylate (PMMA) is widely used as a denture base material, its disadvantages include low strength and low thermal conductivity. The effects on thermal conductivity, flexural strength, thermal diffusivity, and elastic modulus of adding Al_2O_3 and SiC powders in different volumes to PMMA were investigated. A total of 60 specimens were prepared in 10 groups (five groups for the thermal conductivity test and five groups for the flexural strength test (n:6). The specimens were immersed in water for 30 days before the testing. Thermal conductivity values were measured by the transient hot bridge (THB) method, and flexural strengths were measured by the 3-point bend test. A significant difference was found in thermal conductivity, flexural strength, thermal diffusivity and elastic modulus values between independent groups (P <0.001) using the Kruskal-Wallis test. The Kruskal Wallis 1-way ANOVA was used for the post hoc tests after Kruskal Wallis (α=.05). The thermal conductivity of PMMA increased significantly with the addition of 15% SiC and 15% Al_2O_3. The flexural strength values decreased significantly with the addition of 10% SiC and 15% Al_2O_3. The thermal diffusivity values increased significantly with the addition of 10% and 15% SiC. The Young modulus of PMMA decreased when 10% SiC, 10% Al_2O_3 and 15% Al_2O_3 were added. Environmental scanning electron microscope (ESEM) showed that ceramic powders were dissipated in PMMA. The addition of 15% SiC powders to PMMA increased thermal conductivity without significantly reducing flexural strength. This study helped determine the optimum volumes for the use of SiC and Al_2O_3 powders. Knowledge of the importance of this variable will help in more effective modification of denture base resin with SiC and Al_2O_3 powders to improve heat transfer without adversely affecting strength.*

Keywords: *flexural strength, thermal conductivity, polymethylmethacrylate*

1.Introduction

Although no ideal material has been found, PMMA is the most commonly used denture base material [1]. PMMA has limitations, including poor thermal conductivity, leading to reduced taste perception in denture warers as food temperature has been shown to affect taste perception [2-4].When hot or cold food is ingested, the temperature of the oral mucosa changes for a few seconds to between 0 and 70°C and then returns to the normal physiological temperature. In in elderly individuals whose palate mucosa is partially or completely covered with PMMA denture base material, temperature perception is impaired and can affect satisfaction with the prosthesis. A denture base with high thermal conductivity would protect the health of the tissues, [5,6] provide better taste [5,7] and reduce the feel of the prosthesis as a foreign body [5-8]. Different materials with high thermal conductivity can be added to improve the thermal conductivity of PMMA. Silver, aluminum and copper powders have been added to conventional denture base material to improve thermal conductivity. To increase heat transfer by 4.5 times, an addition of 25% by volume is required, but this addition reduces the tensile strength by 35% [5]. Thermally conductive ceramics may be more suitable than metal powders as additives, as most of these ceramics have a thermal conductivity similar to that of metals [9]. In addition, ceramic fillers have a low density, so the weight of the prosthesis is not significantly increased.

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The thermal conductivity of silicon carbide (SiC) exceeds that of copper, alumina (Al_2O_3) and aluminum nitrate (AlN) [10] and is very hard, thermally stable, and of low density [11]. Because it contains a high covalent bond, it is more durable than oxide ceramics, with good thermal properties, good biocompatibility and excellent cytocompatibility, making it suitable for use in medical implants and prostheses [12]. Alumina has been used for reinforcing acrylic resins [13] and is the strongest and most rigid of the oxide ceramics [14]. It has wide application because of its exceptional hardness, excellent dielectric properties, good heat resistance and thermal properties [14].

Heat conduction in solids is defined according to Fourier's law as the rate of heat transfer through a unit thickness of the material per unit area per unit temperature difference according to the following equation [15]:

$$\dot{Q}_{cond} = -kA\frac{dT}{dx} \qquad [W]$$

In order to improve heat transfer, either a material with high thermal conductivity should be selected or the thickness should be reduced. Since it is not always possible to reduce or increase the thickness, material selection and consequently thermal conductivity gains importance [15]. Thermal diffusion also includes time. The loss of time and temperature spent by heat passing through the prosthetic base is effective in heat diffusion and is related to the thickness of the base [16]. Thermal diffusivity, defined as the ratio of the transmitted heat to the stored heat (volumetric), [17] measures the heat transfer rate of a material from the hot end to the cold end. Therefore, thermal diffusion (thermal distribution) is thought to be the most relevant factor for patients to feel intraoral temperature changes and is considered a measure of thermal inertia [18].

Studies that have attempted to increase the thermal conductivity of PMMA are limited, [5,9,14,19,20] and new approaches are needed. In our previous study, [19] electron microscopy images showed that SiC and Al_2O_3 powders added to PMMA were homogeneously dispersed, increasing thermal conductivity without adversely affecting flexural strength. Therefore, in the present study, we aimed to add these powders in different proportions by volume and to evaluate them with a different thermal conductivity measurement technique. In addition, we aimed to investigate the thermal diffusivity and elastic modulus of the modified PMMA. The hypothesis of the study was that the thermal conductivity, thermal diffusivity, elastic modulus and flexural strength of polymethylmethacrylate would be altered by the addition of fillers.

2.Materials and methods

Stainless-steel molds were made for the 3-point bend test in the form of a rectangular prism [14,21,22] of 3 mm in thickness, 10 mm in width and 65 mm in length (30 specimens in 4 experimental groups and 1 control group) in accordance with the ISO 1567 standard [21]. For the thermal conductivity specimens, stainless steel molds were prepared in a CNC lathe in the form of 60×50-mm and 3-mm-thick rectangles (30 specimens in 4 experimental groups and 1 control group) in accordance with ASTM E1530-06 [23] and were placed in muffles using Type II hard gypsum (Moldano, HerausKulzer, Germany) under a hydraulic press (Carlo de Giorgi, Milan, Italy) with a pressure of 14 MPa (2030 psi) for 30 min. After the gypsum had hardened, the muffles were opened, the molds were removed with a spatula, and the cavities were coated with a separating material (Vertex, Vertex Dental). Conventional heat-polymerized veined pink acrylic resin prosthetic base material (Meliodent, KulzerGmbH, Hanau, Germany) was prepared as10% by volume of acrylic resin powder measured with a balance with an accuracy of 1/10000 g. The weighed powders (Table 1) were dehydrated in a thermostatic oven at 800°C for 2 h, and silane was applied to improve the bond of the powders to the polymer matrix. In the silanization process, 70% ethanol solution was prepared for each powder using distilled water and ethanol. The *p*H was adjusted to 4.5 using acetic acid controlled with a pH meter [24]. Five drops of Clearfil ceramic primer (0.6 g) and 5 drops of GC metal primer II (0.8g) were added (Table 2) and allowed to hydrolyze and silanize for 5 min before the additive powder was added and mixed. After the

powders had dried in an oven at 62°C for 1 h, the additive powders were mixed thoroughly with the PMMA powder in a vortex mixer (finevortex, fluxana), and then methylmethacrylate liquid was added. Once the acrylic resin had reached a doughy consistency that would not stick to the sides of the mixing vessel, it was placed in the mold cavity and the muffles were closed. The hydraulic press was kept under pressure at 14 MPa (2030 Psi) for 5 min, and samples were taken to the dental flask press. Pressed flasks were allowed to stand for 8 h in 74 ± 1ºC water in a constant temperature adjustable water bath and then boiled for 2 h. The specimens were polished using 320-, 600-, and 1200-grit abrasive paper. Sixty specimens were prepared, 30 specimens for the flexural test (5 group and 6 samples for each group) and 30 for the thermal conductivity test. Control and experimental groups are shown in Table 3.

Table 1. Materials used

Name	Manufacturer	Product no	Formula and abbreviation	Density g/mL	Particle Size
Meliodent	Kulzer GmbH	13FEB069	PMMA	d=1.18	121.2 μm
Aluminum oxide powder	Sigma-Aldrich	Lot#MKBH8890V	Al_2O_3	d =3.97	10 μm
Silicon carbide powder	Sigma-Aldrich	Lot#MKBN2849V	SiC	d=3.22	<37 μm

Table 2. Silanes used

Silane	Manufacturer	Product no	Content
Clearfil ceramic primer	Kuraray med. Co.Ltd.	Lot C40004	MTS, MDP, ethanol
GC metal primer II	GC Corp	Lot 1305022	MEPS, MMA

MEPS: thiophosphoric methacrylate, MMA: metylmethacrylate, MTS: 3- methacryloxypropytrimethoxysilane, MDP: 10-methacryloxydecyl dihydrogen phosphate[102]

Table 3. Control and experimental groups

Group number	N	V%
1	6	100% PMMA (control)
2	6	10% SiC, 90% PMMA
3	6	10% Al_2O_3, 90%PMMA
4	6	15% SiC, 85% PMMA
5	6	15% Al_2O_3, 85% PMMA

Flexural strength was measured with the 3-point bend test on the Instron device (model 3344 Instron Ltd., USA). The specimens were immersed in water for 30 days before the test (piston head speed of 1 mm/min, distance between the supports 50 mm). The load was applied until the specimen fractured, with the flexural strength calculated according to the formula:

$$(S) = 3FL / 2BH^2,$$

where S = flexural strength (N / mm^2 = MPa), F = load at fracture (N), L = distance between supports (50.0 mm), B = specimen width (mm), and H = specimen thickness (mm).

The elastic modulus was calculated as follows:

$$E = \frac{FL^3}{48fI},$$

where f is maximum displacement [mm] and I is inertia moment [mm^4]. Inertia moment is calculated from:

$$I = \frac{BH^3}{12}.$$

Thermal conductivity was measured in a physiological temperature range corresponding to possible food and beverage temperatures using the transient hot bridge (THB) method (Figure 1). LINSEIS THB-100 was used for measurement. This method is suitable for measuring the heat conduction of polymer materials [25]. While measuring the thermal conductivity of the prepared samples in the THB-100 tester, a sensor was placed between two flat faces of the two samples and pressed by hand screwing. A controlled heating power of 50 mW was determined and a temperature change proportional to the thickness of the material was determined.

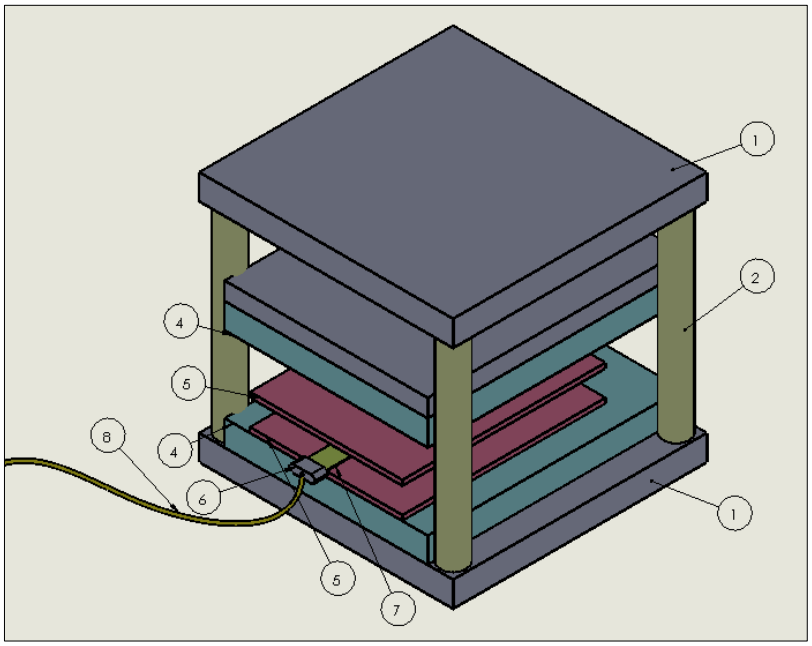

Figure. 1 THB-100 Thermal conductivity measurement device
1: Top and base plates; 2: support system; 4: linings;
5: specimens; 6: jack; 7: probe; 8: cable

Thermal diffusivity was calculated as follows [15]:

$$\alpha = \frac{conducted\ heat}{stored\ heat} = \frac{k}{\rho C_p}[m^2/s],$$

where k: coefficient of heat conduction [W / mK]; ρ: density [kg / m^3] and Cp: specific heat (A measure of the thermal energy storage ability of materials) [J / kgK].

The Kruskal-Wallis post hoc test was used to compare the mean values between the groups (α=.05). The specimens were also examined with an environmental scanning electron microscope (ESEM) to determine the dispersion of the powders added to the PMMA.

3. Results and discussions

The mean and standard deviation values of the data of the current study are given in Table 4. The mean thermal conductivity values of the control group were statistically significantly lower than those of experimental groups 15% SiC and Al_2O_3. The flexural strength of the control group was statistically significantly higher than that of experimental groups 3 and 4, which remained below the minimum recommended value (65MPa) in the ISO 1567 standard [21]. The thermal diffusivity values of the control group were statistically significantly lower than those of groups 3 and 5 (SiC added groups). The elastic

modulus values of the control group were statistically significantly higher than those of groups 2, 3, and 4.

Table 4. Statistical analysis results

	Mean ± Standard Deviation			
	α [mm^2/s]	S[Mpa]	k[W / mK]	E[Mpa]
Group 1	$0.12±0.001^a$	$87.4±6.2^c$	$0.21±0.002^a$	$2313±186^c$
Group 2	$0.13±0.002^b$	$67.9±6^a$	$0.27±0.005^b$	$1116±62^a$
Group 3	$0.14±0.002^c$	$62.1±1.9^a$	$0.27±0.003^b$	$1060±47^a$
Group 4	$0.14±0.002^c$	$60.8±2.4^a$	$0.29±0.004^c$	$1058±51^a$
Group 5	$0.15±0.001^d$	$77.2±8.2^b$	$0.31±0.002^d$	$1360±151^b$
	p< 0.001	p< 0.001	p< 0.001	p< 0.001

In this study, the effect of different ceramic powders on the thermal conductivity of a denture base material was investigated. The first hypothesis of our study, an increase in the thermal conductivity of the 4th and 5th groups is supported. However, the second hypothesis, an increase in flexural strength, was rejected. Covering the palate with a denture base has been reported to reduce sensory function and mastication [26, 27]. In in vivo studies, some patients reported that covering the palatal mucosa with a prosthetic base had a negative effect on their sense of taste, and designs with an open base were developed [28, 29]. However, when the palate is less covered by a prosthesis, retention is decreased [30].

Specimens with different thicknesses and shapes have been used to determine the thermal conductivity of polymers [5,9]. In our study, 60×50×3 mm-specimens were prepared as recommended by the manufacturer [25]. In the ESEM used in our study, specimens can be examined in the environmental pressure mode without having to be coated with a conductive material and without disturbing the structure or moisture balance. It is therefore a suitable imaging device for polymeric materials.

The midline fracture of a maxillary complete denture is a common clinical problem, [31] leading to studies investigating the flexural strength of prosthetic base materials, [22, 31, 32] typically with a 3-point bend test that reflects the loads that prostheses are exposed to during clinical use [32]. Therefore, in this study, we investigated the flexural strength of modified acrylic resin with this method.

Aluminum oxide (Al_2O_3) has been used to reinforce PMMA, [13] with Ellakwa et al. [14] reporting that thermal conductivity and flexural strength increased with Al_2O_3 additions, leading to increased patient satisfaction [14]. Similarly, in our study, thermal conductivity was increased with the addition of Al_2O_3, but there was no difference in flexural strength. In a recent study, Al_2O_3 added to a soft lining material increased thermal conductivity and diffusivity [33].

The shape and size of the particles added to the PMMA are important in terms of thermal conductivity. Spherical particles of Al_2O_3 have been reported to significantly increase heat diffusion and flexural properties because of the distribution of alumina spheres in the powder [14]. In our study, spherical powders were used. The average particle size of the fillers allows maximum loading of the PMMA, [8] the particle size is important as thermal conductivity depends on the filler resin ratio [5].

Similarly, in our previous study, the group containing aluminum oxide powder with a particle size of 10 μm was one of the best in terms of both flexural strength and thermal conductivity. The SiC group with a particle size of 37 μm had the best flexural strength. However, in the experimental groups (nanoSiC and nano HA) containing smaller particle sizes, especially with nano-size powders, the flexural strength was reduced. Both the thermal conductivity and flexural strength values of the PMMA containing nano-size SiC filler were significantly lower than those of the group containing the micro-size SiC filler [19].

Yadav and Elkawash [34] reported that the addition of 5% Al_2O_3 to the acrylic resin resulted in reduced flexural strength because of non-homogeneous dispersion of the filler particles in the acrylic resin, which led to increased stress. They also recommended silane application.

As SiC is a highly biocompatible material, SiC nanoparticles have been recommended for different technological applications. The addition of SiC has been reported to increase the thermal conductivity of denture base materials without changing the flexural properties [35]. Four studies [12,36-38] have reported adequate biocompatibility; however, two studies [39, 40] reported insufficient biocompatibility. This difference may be because of different sintering parameters, surface properties or chemical impurities [12].

The fillers used in our study were selected because of their high thermal conductivity, low density, high strength and biocompatibility and were added to the PMMA at a rate of 10% by volume. In the study of Yadav et al. [20] participants who had 20% aluminum particles added to the palate of their maxillary complete denture stated that it increased the feeling of hot and cold and they preferred this new prosthesis. In another study, nano-sized silver particles were reported to increase the thermal conductivity of the denture base resin and could be used in the palatal region of the prosthesis [41]. Similar results were found in another recent study using gold nanoparticles [42].

Silane containing 3-methacryloxypropyltrimethoxysilane (MTS) has been reported to increase the connection of ceramics and resin cements [43-45]. If combined with thiophosphoric methacrylate (MEPS) and MTS, it can improve the bonding of resin to various restorative materials (precious metal alloys, non-precious metal alloys, silica-based ceramics and resin composites) [43].

4.Conclusions

The addition of 15% SiC and 15% Al_2O_3 ceramic powders increased thermal conductivity. However, 15% Al_2O_3 decreased the flexural strength of PMMA. 15 % SiC did not decreased the flexural strength of PMMA.

According to the results of our study, we can recommend that 15% by volume of SiC filler powder be placed in the palatal region of the prosthesis to increase thermal conductivity without reducing the durability of PMMA or increasing its weight.

ESEM images show that the added powders were homogeneously dispersed in the PMMA matrix and that there was good bonding between the two substances (Figure 2-6).

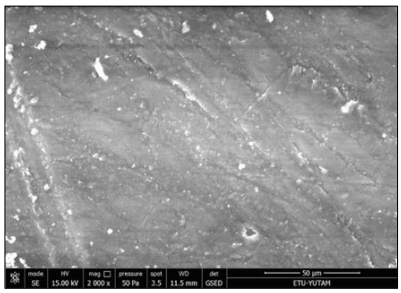

Figure 2. ESEM (Quanta FEG-250) image of the control group at ×2000 magnification shows a homogeneous and porous structure. This porous structure is one of the reasons for the low thermal conductivity [29]

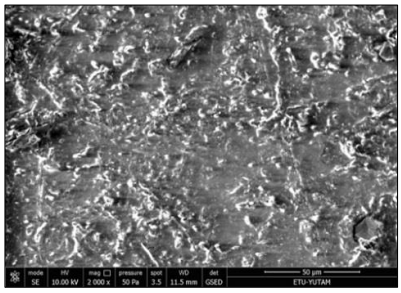

Figure 3. ESEM image of 10% SiC group at ×2000 magnification shows that this distribution does not create continuous transmission paths for heat conduction

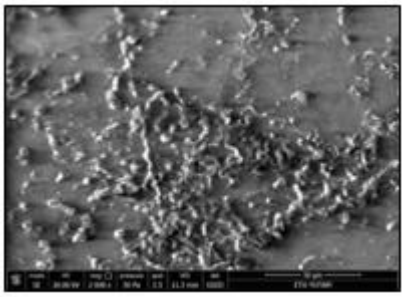

Figure 4. ESEM image with ×2000 magnification of 10% Al_2O_3 group shows that this distribution does not create continuous transmission paths for heat conduction

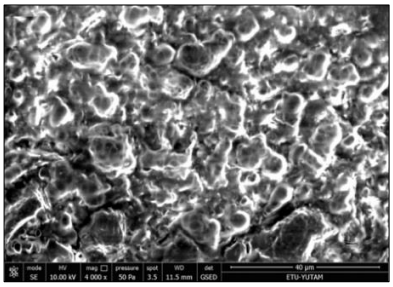

Figure 5. ESEM image of 15% SiC group at ×4000 magnification shows that the porous regions of PMMA are filled by SiC and provide excellent integration with PMMA. This may explain the fact that there is no difference from the control group in terms of three flexural strength values. This distribution also creates continuous transmission paths for heat conduction

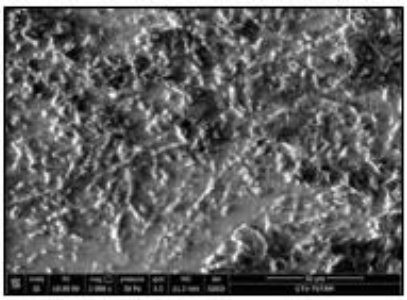

Figure 6. In the 15% Al_2O_3 group ×2000 magnification ESEM image, the filling powders were distributed homogeneously in the matrix explaining the increase of thermal conductivity

However, more homogeneous dispersion of filler powders in the PMMA like alloying with PMMA should be studied.

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