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Evaluation of the Effect of Addition of Aluminium Oxide on Thermal Diffusivity and Flexural Strength of Heat Polymerized Acrylic Resin

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Abstract

Aims and Objective: Complete denture wearers usually complain of poor taste perception as the palate is completely covered with the denture base which has poor thermal characteristics. The purpose of this study was to compare the thermal diffusivity and flexural strength of heat polymerized Polymethylmethacrylate (PMMA) denture base resin with the addition of different concentrations of aluminum oxide particles of 100 µm size. This study also aims to determine whether addition of aluminum oxide particles to denture base resins in any concentration should be recommended or not considering various pros and cons.

Materials and Methods: Heat polymerized PMMA was used to fabricate cylindrical specimens (1 cm in diameter and 1 cm in length) for thermal diffusivity testing with K type thermocouple embedded in the center and rectangular specimens for determining flexural strength. In addition to specimens with 5%, 10% and 20% concentrations of Al_2O_3 particles, control specimens were also fabricated without any reinforcement. One at a time cylindrical specimens were immersed in the thermostatic bath maintained at 70ºC+1ºC and transient temperature per second (T) was recorded which was used to calculate the thermal diffusivity values. Rectangular Specimens with the above stated aluminium oxide loadings were fabricated and were then tested for flexural strength by using 3-point bending test in Lloyd's Universal Testing Machine at 5mm/minute crosshead speed. The readings thus obtained for thermal diffusivity and flexural strength were subjected to statistical analysis using one-way ANOVA and Tukey's Multiple Comparison Test.

Results: Mean thermal diffusivity values of heat polymerized acrylic resin increased with the increasing amount of its reinforcement with aluminium oxide by weight as compared to the control group. Testing of heat cured acrylic resin rectangular specimens for flexural strength depicted that mean flexural strength decreased significantly with the increasing amount of its reinforcement with aluminium oxide by weight.

Conclusion: Although with the addition of Al_2O_3 particles to PMMA resin significantly increased thermal diffusivity which would lead to better taste perception in denture wearers, longevity of the denture base cannot be compromised. Hence, we need to eliminate possible reasons of decreased flexural strength of PMMA resin with addition of aluminium oxide particles before recommending its clinical use.

Keywords: K type thermocouple, thermal diffusivity, aluminium Oxide, flexural strength

Introduction

Resin polymers are the most commonly used denture base material in Prosthodontics. Introduced in 1937 by Dr. Walter Wright, use of PMMA still continues because of its favorable working characteristics. Despite its popularity, it lacks some of the beneficial properties like thermal diffusivity and flexural strength.

Tissues in the oral cavity are continuously exposed to the transient thermal changes occurring during intake of various foods and liquids which influence the taste of certain foods which is usually perceptible through the palatal area. In complete denture wearers, as the palate is completely covered by the denture base, the thermal characteristics of the denture base material will determine that transient temperature changes to be transmitted to the underlying tissues of the palate. Thus, although often overlooked, thermal diffusivity of denture base material is an important factor in determining patient's satisfaction[1].

Thermal diffusivity can be defined as a measure of transient temperature changes[1,2].In the present study, thermally conducting ceramics in the form of aluminium oxide particle of size 100 micrometers is used in an attempt to improve thermal diffusivity of denture base material.

To develop a denture base material with good thermal diffusivity, its strength must not be neglected which is essential for its longevity. Most fractures of the dentures occur inside the mouth during function, majorly because of resin fatigue. Flexural fatigue occurs after repeated flexing of a material which occurs due to the development of microscopic cracks in areas of stress concentration. With continued loading, these cracks fuse to an ever-growing fissure that gradually weakens the material. Final loading cycle exceeding the mechanical capacity of the remaining sound portion of the material leads to catastrophic failure. So, the potential impact of aluminium oxide on the flexural strength, which might tell us about how the denture base resin would hold up under function, needs to be assessed.

Therefore the present study, 'Evaluation Of The Effect Of Addition Of Aluminium Oxide On Thermal Diffusivity And Flexural Strength Of Heat Polymerized Acrylic Resin' evaluated the thermal diffusivity in the temperature range of 0º-70ºC which corresponds to the temperature of meals ingested. The flexural strength of heat polymerized acrylic resin after adding different percentages of Al_2O_3 was also evaluated.

Materials and Methods

The specimens for this study were prepared at the Department of Prosthodontics, Government Dental College, Amritsar by using two detachable brass dies. A total of 80 test specimens of heat polymerized PMMA (Pyrax) were fabricated and tested which were divided into 2 group as under:

Group A: 40 cylindrical test specimens for thermal diffusivity testing in Department of Biotechnology at Guru Nanak Dev University, Amritsar.

Group B: 40 rectangular test specimens for the measurement of flexural strength at Central Institute of Plastics Engineering and Technology, Amritsar.

Each group was further divided into 4 subgroups [Figure 1,2(a),2(b)] of 10 specimens each as under:-

Group AI & BI: Specimens were prepared from heat cured PMMA powder and liquid without any reinforcement which served as control

Group AII & BII: Specimens were prepared from heat cured PMMA with 5% reinforcement of aluminium oxide by weight

Group AIII & BIII: Specimens were prepared from heat cured PMMA with 10% reinforcement by weight

Group AIV & BIV: Specimens were prepared from heat cured PMMA with 20% reinforcement of aluminium oxide by weight

Aluminium oxide (100µm particle size, Qualigens, Thermo Fisher Scientific India Pvt, Ltd., Mumbai)) was added to the polymer of heat polymerized acrylic resin (Pyrax, Pyrax Polymers, India) in different loadings by weight after measuring with a electronic digital balance [Figure 3] and then was mixed with Spinix mixing machine [Figure 4]

For Thermal Diffusivity Testing

40 cylindrical test specimens (Group A) of heat cured PMMA resin (1 cm in diameter and 1 cm in length) were prepared by placing the material in the first die [Figure] with the above mentioned loadings of aluminium oxide of various subgroups.

This die consisted of three compartments [Figure 5(a),5(b)]

Lower: It could be separated into two halves for the easy removal of samples after curing and had the provision for receiving four cylindrical specimens at a time.

Middle: It had two parts. First one was such that it helped in creating space for the thermocouple wires. Its second part consisted of two halves which has the provision for the thermocouple wires exiting from one face of the cylindrical specimens.

-Upper: It is fitted over the whole assembly and was tightened with screws to cover the thermocouple wires.

Packing of heat cure acrylic resin of different loadings of aluminium oxide in the lower compartment of die was done. After that, first part of the middle compartment of the die was placed over it to create space for the placement of thermocouple wires when still in the dough stage and then it was removed. Subsequently, K type thermocouple wires were placed along with the bead junction exactly in the center of the test specimens. Next, Second part of the middle compartment was placed over the lower compartment providing space for the exiting thermocouple wires from the specimens. Finally, upper compartment was screwed tightly.

After curing, all test specimens were conditioned at room temperature in distilled water for 24 hours before measurement and equilibrated in a 0+1ºC (T_0) for 30 minutes before testing. The specimens were then immersed in a thermostatic bath at 70+1ºC[Figure 6]and the thermocouple wires exiting the face of the cylindrical specimen were attached to the digital PID controller (digital manufacturers, TAIE Taiwan)[Figure 7]which was further attached to the laptop (Sony VAIO E series VPCEA23EN with XP windows)[Figure 8] with the help of universal convertor KA 301 (serial signal convertor along with RS-485, TAIE Taiwan) [figure 9] Universal convertor helped in converting the digital signals into analog signals and the temperature(T) readings at the centre of the specimens were recorded per second for up to 10 minutes. Temperature (T) was converted to a normalized temperature (T_n) according to the equation: -

$T_n = (T - T_s) / (T_0 - T_s)$

Where T = Transient temperature recorded per second for up to 10 minutes at the centre of the specimen after placing it in a thermostatic bath.

T_s = Surrounding temperature of the specimen after placing it in thermostatic bath i.e. 70+1ºC

T_0 = Temperature at which specimens were equilibrated before placing them in thermostatic bath i.e. 0+1ºC

Natural log of T_n (ln T_n) values were calculated. Graph was plotted between time and ln T_n for each specimen. Using linear least square fits, slopes of the graphs (S) were obtained which were then used to calculate thermal diffusivity for each specimen (Δ) according to the following equation:

$\Delta = -S/\{(5.7832/R^2) + (\pi^2 /L^2)\}$

Where S = slope obtained from the graph

R = Radius of the cylindrical specimen (5 mm)

L = Length of the cylindrical specimen (10 mm)

Thermal diffusivity was measured in millimeter square per second.

The data obtained was compiled, tabulated and subjected to statistical analysis by using one way ANOVA and Tukey's Multiple Comparison Test.

For Flexural Strength Measurements

40 rectangular test specimens (Group B) of heat cured PMMA resin (65 mm x 10 mm x 3.3 mm) were fabricated using second die [Figure 10] with previously mentioned aluminium oxide loadings of various subgroups by packing and curing the material in dough stage. All specimens were stored in water at 37ºC for 24 hours and then measurements for flexural strength was done by using 3 point bending test in Lloyd's Universal Testing Machine [Figure 11] at 5 mm/minute crosshead speed. The flexural strength of each specimen was determined as follows:

$S = 3WL/2bd^2$

Where:

S = Flexural Strength

W = Peak Load

L = Distance between supports (50 mm)

b = Width of the specimen (10 mm)

d = Specimen thickness (3.3 mm)

The readings, thus obtained, were subjected to statistical analysis using one-way ANOVA and Tukey's Comparison Test.

Results

With increasing amount of reinforcement of PMMA resin with Al_2O_3 mean thermal diffusivity values are increased [Table 1] and mean flexural strength values are decreased [Table 2]. Analysis of thermal diffusivity values and flexural strength values for study group A and group B by one-way ANOVA indicated a very highly significant difference between the means of subgroups AI to AIV. (F

= 34.18, P = 1.24 x 10^{-10}) [Table 3] and subgroups BI to BIV (F = 165.7, p < 2 x 10^{-16}) [Table 4]

Since the number of subgroups were more than two, therefore to determine as to which of the means of groups were significantly different, the observed differences between each of the possible pairs among the groups were subjected to Tukey's Multiple Comparison Test [Table 5, 6].

SPECIMEN NUMBER	THERMAL DIFFUSIVITY (In millimetre square per second)			
	GROUP-AI	GROUP-AII	GROUP-AIII	GROUP-AIV
1.	0.103271	0.102180	0.113146	0.122749
2.	0.081005	0.112268	0.109026	0.125324
3.	0.103877	0.105391	0.120174	0.129353
4.	0.100453	0.103998	0.112134	0.122113
5.	0.105028	0.113177	0.108693	0.117054
6.	0.096970	0.113358	0.117054	0.127960
7.	0.100484	0.108966	0.112328	0.124809
8.	0.106058	0.119235	0.11866	0.128505
9.	0.106270	0.108239	0.117812	0.142168
10.	0.099454	0.113752	0.115418	0.128384
MEAN	0.100286725	0.110056388	0.11444456	0.126842032

Table 1: Tabulated data of thermal diffusivity of group A

SPECIMEN NUMBER	MAXIMUM FLEXURAL LOADS (In MPa)			
	GROUP-BI	GROUP-BII	GROUP-BIII	GROUP-BIV
1.	99.87	61.00	33.32	40.00
2.	127.42	68.65	50.50	41.18
3.	103.47	84.4	36.65	38.00
4.	90.27	82.16	37.19	38.00
5.	94.21	73.61	37.35	33.47
6.	112.2	73.00	48.93	30.33
7.	96.66	69.00	49.51	40.42
8.	106.8	80.00	42.16	31.93
9.	95.67	84.00	44.45	35.9
10.	105.1	70.64	47.93	34.12
MEAN	103.167	74.646	42.799	36.335

Table 2: Tabulated data of flexural strength of group B

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F-Value (Variance ratio)	P value for F
Main Groups	3	0.003639	0.0012132	34.18***	1.24×10^{-10}
Residuals	36	0.001278	0.0000355		
Total	39	0.004917			

Table 3: One way ANOVA table for thermal diffusivity

*:Significant (p < 0.05), **: Highly Significant (p < 0.01), ***: Very Highly Significant (p <0.001), NS: Non significant (p > 0.05)

Source of Variation	Degrees of Freedom	Sum of Squares	Mean Square	F-Value (Variance ratio)	P value for F
Main Groups	3	28620	9540	165.7***	$< 2 \times 10^{-16}$
Residuals	36	2073	58		
Total	39	30693			

Table 4: One way ANOVA table for flexural strength.

*:Significant (p < 0.05), **: Highly Significant (p < 0.01), ***: Very Highly Significant (p <0.001), NS: Non significant (p > 0.05)

Comparison among groups	Difference	Lower	Upper	p value	Significance level
AII-AI	0.009769663	0.002593583	0.01694574	0.0041949	**
AIII-AI	0.014157931	0.006981851	0.02133401	0.0000330	***
AIV-AI	0.026555307	0.019379227	0.03373139	0.0000000	***
AIII-AII	0.004388268	0-0.002787812	0.01156435	0.3660938	NS
AIV-AII	0.016785644	0.009609564	0.02396172	0.0000016	***
AIV-AIII	0.012397376	0.005221296	0.01957346	0.0002435	***

Table 5: Tukey's multiple comparisons of means of thermal diffusivity values at 95% family wise confidence level (using Bonferroni transformations for p value)

*: Significant (p < 0.05), **: Highly Significant (p < 0.01), ***: Very Highly Significant (p <0.001), NS: Non significant (p > 0.05)

Comparison among groups	Difference	Lower	Upper	p value	Significance level
BII-BI	-28.521	-37.65982	-19.382184	0.000000	***
BIII-BI	-60.368	-69.50682	-51.229184	0.000000	***
BIV-BI	-66.832	-75.97082	-57.693184	0.000000	***
BIII-BII	-31.847	-40.98582	-22.708184	0.000000	***
BIV-BII	-38.311	-47.44982	-29.172184	0.000000	***
BIV-BIII	-6.464	-15.60282	2.674816	0.244002	NS

Table 6: Tukey's multiple comparison of means of flexural strength values at 95% family wise confidence level (using Bonferroni transformations for p value)

*:Significant ($p < 0.05$), **: Highly Significant ($p < 0.01$), ***: Very Highly Significant ($p < 0.001$), NS: Non significant ($p > 0.05$)

Discussion

High thermal diffusivity of denture bases will lead to improved tissue health[3,4], better appreciation of taste[4] and reduces the foreign body feeling of the dentures[5].

Thermal conductivity measurements are taken under steady state temperature conditions whereas thermal diffusivity of denture base affects the ability to transmit thermal changes from the oral cavity to the underlying tissues of the palate. Therefore, considering the patient's experience with intraoral temperature changes, thermal diffusivity is the most relevant parameter.

Perception of taste is significantly affected by food temperature. **Kapur, KK & Fischer, EE (1981)[4]** conducted a study in which denture base conductivity was found to have an important effect on parotid gland secretions, which have been shown to be a good approximation of gustatory sensitivity. It was found that altered gustatory response was associated with sustained high palatal temperatures.

Heat and cold influence the taste of certain foods which is usually perceptible through the palatal area. The differences between the relative abilities of natural and artificial dentition patients was studied by **Giddon, DB et al (1954)[6]** for the sweetness of solid foods. They found that denture patients were less capable of differentiating between sweetness of different groups of solid foods as their palates were covered with denture bases.

Despite several efforts were being made to improve thermal characteristics of denture base materials, none of them were without major shortcomings. Some of them worth mentioning includes replacement of acrylic with metal as dental bases like aluminium, gold, cobalt and chromium alloys as done by **Halperin, AR (1980)[3]**. Others include addition of metal filler particles like that of silver, copper and aluminium as done by **Sehajpal, SB & Sood, VK (1989)[5]**.

As alternative to metallic powder fillers, thermally conducting ceramics such as sapphire (single crystal form of Al_2O_3), silicon nitride (Si_3N_4) and aluminium nitride (AlN) may be useful for increasing the thermal diffusivity. Benefit of using ceramic fillers is the lower filler density which retains the light weight of the acrylic denture bases. Moreover, as these ceramics powders are white in color, it is less likely to alter the finished appearance of the denture base material. In the present study aluminium oxide particles of 100 µm added to heat polymerized acrylic resin (Pyrax) to achieve loadings of 5%, 10% and 20% by weight.

The temperature extremes produced in the oral cavity by hot and cold liquids was studied by **Palmer, DS et al**

(1992)[7] which suggested a range of 0º - 67º C appropriate for testing thermal changes in dental materials. In the present study, measurement of thermal diffusivity was done within physiological temperature range bearable by the oral tissues i.e. 0º-70º C.

The present study has used the experimental procedure on the basis of the method formulated by **Watt, DC & Smith, R (1981)**[8] but with modifications of capturing data through a personal laptop. This modified method allowed accurate and rapid determination of thermal diffusivity by using specimens of cylindrical geometry (10mm in diameter and in length each) that contain an embedded thermocouple for temperature measurements within the sample.

Thermal diffusivity measurements of all study groups were statistically analyzed using one way ANOVA test and then with Tukey's Multiple Comparison Test to make all valid comparisons.

Mean thermal diffusivity values of heat polymerized acrylic resin increased (0.1100, 0.1144 and 0.1268 mm^2/sec)[Table 1] with the addition of increasing amounts of aluminum oxide by weight (5%, 10% and 20% respectively) as compared to the control group (0.1002 mm^2/sec) [Table 1].These observations were in accordance with the study conducted by **Messer smith, PB et al (1998)**[1] who added sapphire whiskers of diameter 3 to 5 µm and lengths of 40 to 60µm which lead to increased thermal diffusivity (14.64×10^{-2} and 16.08×10^{-2} mm^2/sec) of denture base resin.

From Tukey's Multiple Comparison Test [Table 5], it was analyzed that mean thermal diffusivity of Group AII, Group AIII and Group AIV were significantly different from the control group. Also significant difference was found between Group AII and Group AIV & between Group AIII and Group AIV while there was no significant difference between thermal diffusivity values of Group AII and Group AIII.

Percentage increase in the thermal diffusivity was found to be 9.75%, 14.11%, and 26.47% [Bar graph-1] with the addition of 5% (group AII), 10% (group AIII) and 20% (group AIV) aluminium oxide by weight respectively. These results are comparable with the study conducted by **Ellakwa, AE et al (2008)**[2] who demonstrated that alumina addition of 5%, 15% and 20% (by weight) was responsible for 5%, 25% and 30% increase in thermal diffusivity respectively in comparison to reinforced group.

Arora, N et al (2011)[9] conducted a study in which by the addition of sapphire fillers, ability of heat cure acrylic resin to transmit transient temperature changes were increased which was confirmed by the results of the present study.

From the results of the present study, it could be inferred that aluminium oxide formed conductive pathways within the polymethyl methacrylate resin that increased thermal diffusivity when added in higher concentrations.

Not neglecting the strength of denture base resin after reinforcement with aluminium oxide particles, flexural strength was also tested in the present study. From the viewpoint of engineering, a denture can be idealized as simple beam in which maximum stress concentration occurs at the midpoint of its unsupported length. Taking this into account, rectangular specimens were prepared for the transverse bending test, in which specimens were bent until fracture using a three-point flexural test technique. The flexural strength represents the highest stress experienced within the material at its moment of rupture.

Flexural strength of all study groups was also statistically analyzed using one-way ANOVA test and then with Tukey's Multiple Comparison Test to make all valid comparisons.

Decrease in the mean flexural strength values of heat polymerized acrylic resin was seen with the addition of increasing amounts of aluminium oxide (5%, 10% and 20% by weight). It was 73.64, 42.79 and 36.33 MPa, as compared to the control group which was 103.16 MPa [Table 2]. These observations were comparable to the study done by **Yadav, NS & Elkawash, H (2011)**[10] which found that there was significant decrease in flexural strength of acrylic resin reinforced with 5% aluminium oxide irrespective of the technique used for its processing.

From Tukey's Multiple Comparison Test [Table 6], it was analyzed that mean flexural strengths of Group BII, Group BIII and Group BIV were significantly lower than the control group. Also, significant difference was found between Group BII and Group BIII & between Group BII and Group BIV while there was no significant decrease in the mean flexural strength between groups BIII(42.79MPa) and BIV (36.33 MPa).

Percentage decrease in the mean flexural strength was found to be 27.64%, 58.51% and 64.78% [Bar graph-2] with the addition of 5% (group BII, 10%(Group BIII) and 20% (group BIV) aluminium oxide by weight. Hence, the present study also confirmed the results of the study conducted by **Vojdhani, M et al (2012)**[11] who demonstrated that there was significant decrease in mean flexural strength of heat polymerized acrylic resin when it was reinforced with 5% by weight of Al_2O_3.

A study conducted by **Ellakwa, AE et al (2008)**[2] concluded that there was an increase in the mean flexural strength with the increasing concentration of reinforcing Al_2O_3 added to the heat cured resin. Another study done by **Arora, N et al (2011)**[9] also had similar results of significantly higher mean flexural strength of reinforced group on addition of sappire fillers in 25% ratio by weight. In Nutshell, the results of these studies are not in agreement with our study.

Possible reasons for the reduction in the mean flexural strength of heat polymerized acrylic resin after reinforcement with aluminium oxide could be: decrease in the cross section of the load-bearing polymer matrix; stress concentration because of too many filler particles; change in the modulus of elasticity of the resin and mode of crack propagation through the specimen due to increased amount of fillers; non uniform distribution of filler particles, void formation from entrapped air and moisture; incomplete wetting of the fillers by the resin; and the fact that Al_2O_3 acts as an interfering factor in the integrity of the polymer matrix[11].

The present study has limitation for simulating various clinical and physiological conditions such as presence of saliva, exposure to various foods at varying transient temperatures for thermal diffusivity measurements, difference of specimen from actual denture configurations and repetitive mechanical stressing during mastication for strength measurement of heat polymerized acrylic resin reinforced with different loadings of Al_2O_3.

Conclusions

In the present study it was observed that:

1) Heat polymerized PMMA denture base resin specimens reinforced with different concentrations of aluminium oxide (5% Group AII, 10% Group AIII and 20% Group AIV) demonstrated higher thermal diffusivity values (0.1100, 0.1144 and 0.1268 mm^2/sec).

2) Thermal diffusivity of the specimens increased with the increasing concentration of aluminium oxide in it i.e. 9.74%, 14.11% and 26.47% increase with 5%, 10% and 20% by weight addition of aluminium oxide respectively as compared to the unreinforced group.

3) Flexural strength of the specimens with addition of aluminium oxide i.e. 5%, 10% and 20% was lower (73.64, 42.79 and 36.33 MPa respectively) as compared to the control group (103.16 MPa).

4) Flexural strength of the specimens decreased with the increasing concentrations of aluminium oxide in it. The highest decrease in flexural strength was noticed with the Group IV (64.78%) followed by the Group BIII (58.51%) and Group BII (27.64%) i.e. 20%, 10% and 5% by weight addition of aluminium oxide respectively as compared to the unreinforced group (Group BI).

5) Although significant improvement in the thermal diffusivity of heat polymerized PMMA denture base resin was seen with the addition of different concentrations of aluminium oxide which may lead to better taste perception but at the same time decrease in the flexural strength cannot be neglected.

Within the limitations of the present study, conclusions are drawn on the basis of the results obtained. Some other measures are needed to be evaluated, so that addition of aluminum oxide to PMMA does not affect its flexural strength significantly. Then only it could be further used in the future by taking advantage of its improved thermal diffusivity property.

Legend Figures

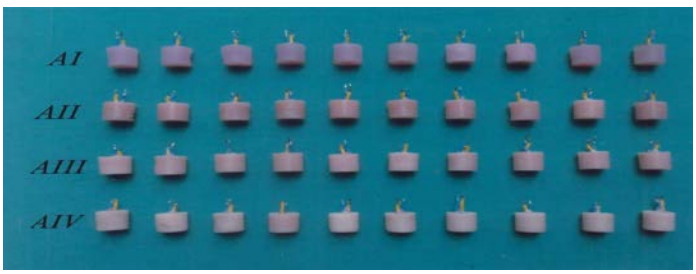


Figure 1: Specimens for thermal diffusivity Testing

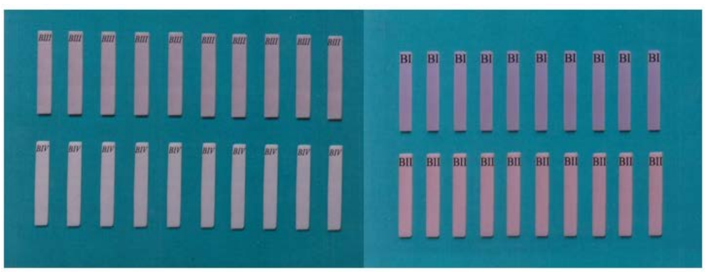


Figure 2: Specimen for measurement of flexural strength.

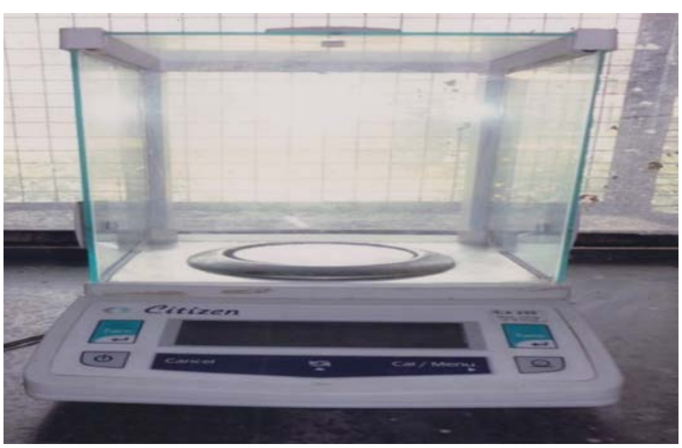


Figure 3: Electronic Digital Balance

Figure 4: Spinix mixing machine

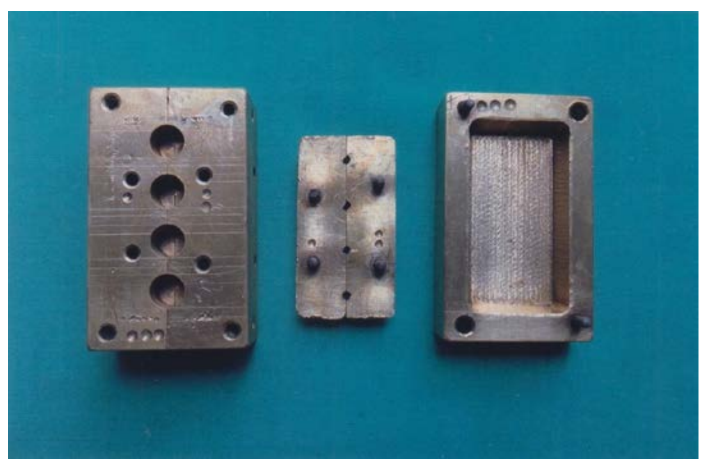


Figure 5: Die for fabrication of thermal diffusivity specimens

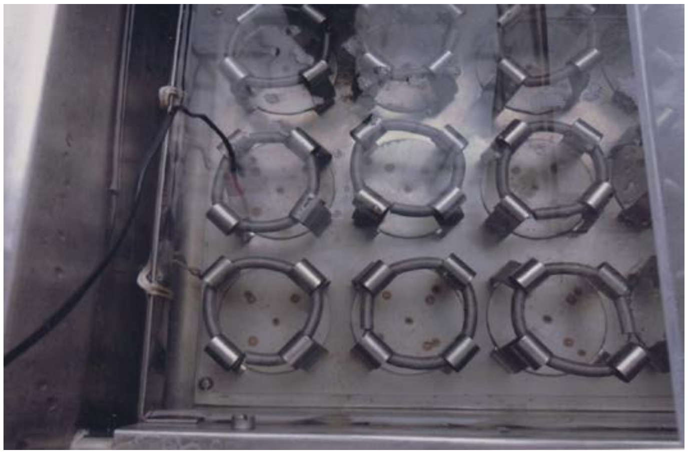


Figure 6:Thermal diffusivity specimen placed in water bath maintained at 70±1ºC

Figure 7: Digital PID Controller (Digital manufacturers, TAIE Taiwan) along with RS-485

Figure 8: Thermal Diffusivity testing set up

Figure 9: Universal converter KA301(Serial digital converter, TAIE, Taiwan) along with wire which connect it with RS 485

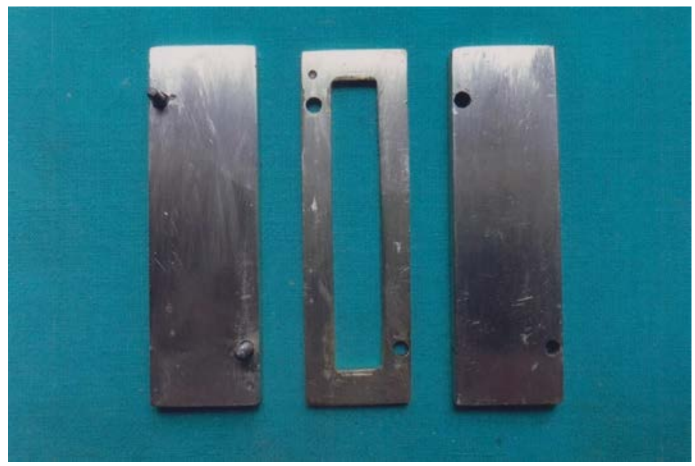


Figure 10: Die for fabrication of specimens for flexural strength testing

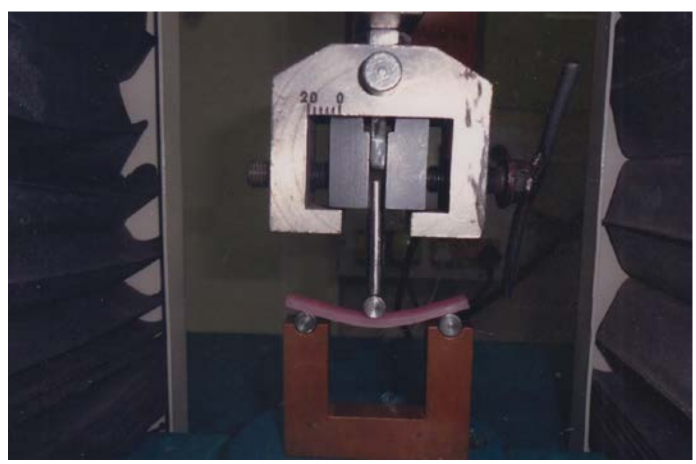
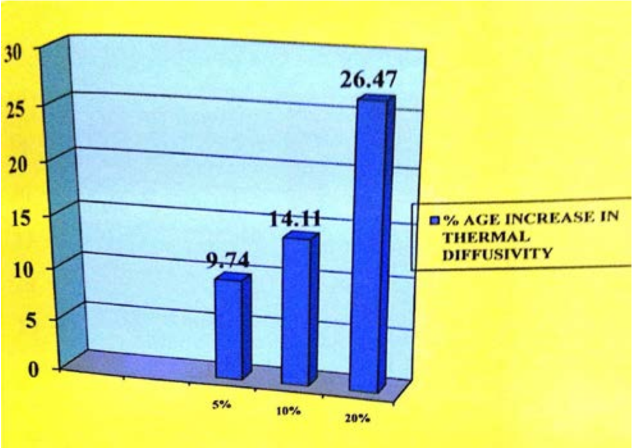
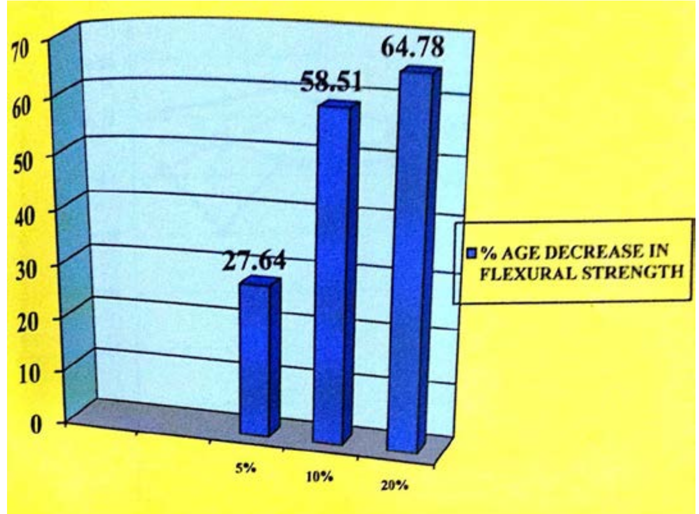


Figure 11: Three point flexural strength test in Lloyd's universal testing machine



Graph 1: Percentage increase in mean thermal diffusivity after addition of different concentrations of aluminium oxide.



Graph 2: Percentage decrease in mean flexural strength after addition of different concentrations of aluminium oxide.

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