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THE EFFECT OF CHEMICAL (GLUTARALDEHYDE) AND MICROWAVE STERILIZATION ON FLEXURAL STRENGTH OF AUTOPOLYMERIZING (PMMA) RESINS.

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ABSTRACT: This Study evaluated the effect of disinfection methods on the Flexural Properties of Auto Polymerizing Resin. (Tooth Colored and Repair Resin). Specimens were exposed to microwaves for 15 min and 2 % Glutaraldehyde for 10 hours. Specimens stored in Water for 12 hours were used as control. For each procedure 10 specimens were used. The result indicated that changes in Flexural Strength observed were of no significance for both Tooth Colored and Repair Resin. The Microwave method is useful alternative to immersion disinfection having advantage of less time consumption.

KEYWORDS: Glass measuring Jar, Micro-wave Energy, Flexural Strength.

INTRODUCTION

Besides the common contagious infections recent increase in the incidence of communicable diseases such as Hepatitis B and Acquired Immuno Deficiency Syndrome (AIDS) has led to a significant change in attitude towards the importance of Cross infection control in dentistry. Because the oral Operating environment includes saliva and frequently a mixture of blood and saliva, the risks from blood-borne viruses such as those causing Hepatitis-B and AIDS are of particular concern¹⁻⁵. Potential sources of transmission of infection from patient to dental technician are impression, impression trays and gypsum casts ^{6, 7}. In addition the dental prosthesis at various stages of trial and insertion can transmit infections from dental staff to the patient. If proper measures are not taken a cycle of cross contamination may occur⁸ exposing the dentist, dental staff, and the patient to transmission and acquisition of disease

Sterilization and disinfection have become the most popular and widely used methods for control of infection^{1,9}. Since many materials used in dentistry cannot be subjected to high heat, or autoclave, chemical agents must be used to sterilize and disinfect them ¹⁰.

The use of disinfectants to sterilize the acrylic resins has been documented $^{11}\,$ and shown to be generally effective. However use of disinfectants may prove time consuming. Sterilization methods used in dentistry include 2,13 -

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1. Heat

- a. Dry Heat (i.e. 160° c for 1 hour)
- b. Autoclave with steam at 121 ° c and 15 psi pressure for 30 minutes.
- c. Unsaturated Chemical Vapour (i.e. at 127 ° c and 25 psi for 20 minutes.
- d. Boiling water for 30 minutes.

2. Chemical

- a. liquid (i.e. immersion in alkaline Glutaraldehyde 2 % for 10 hours, immersion in 1 % solution hypochlorite for 10 hours)
- b. Gaseous (ethylene-oxide).

An alternate method of sterilization for non-autoclavable dental items is receiving some limited but notable research interest. Rohrer and Bulard ¹³ investigated the use of microwave energy for sterilization. They used several types of micro-organisms generally representing intra oral micro flora, as well as variety of dental instruments and prosthesis. They reported that microwave energy could be used for consistent sterilization and that aerobic spores forming micro-organisms, the more resistant to sterilization could be eliminated with a 15 min. microwave energy exposure. They also reported that dentures subjected to microwave energy were dimensionally identical before and after exposure.

Auto polymerizing PMMA resins often used for a variety of applications in restorative dentistry.¹⁴

- Repairs of Fractures/broken dentures.
- Provisional restoration (Temporary crown and bridge work: tooth color).
- Relining of denture base at chair side (with pink colored denture repair resin)Special trays for making sec-impressions.
- Fabrication of temporary and treatment partial dentures ¹⁵.

The sterilization of dentures and denture materials is problem. Often patients who have chronic candidiasis are reinfected from their own dentures. Denture returned from dental lab may be contaminated.

Obviously, thermal methods of sterilization are unsatisfactory. Gas sterilization effective, but in almost all situations, it is impractical or unobtainable. Chlorine treatment ¹³ is effective, however is rejected by the patient because of the bleaching effect on the denture.

Effects of glutaraldehyde and microwave disinfections methods on the dimensional stability, flexural properties and micro hardness of heat cured acrylic denture resin was documented by polyzosis GL: Zissis AJ ¹⁶. They investigated no significant changes

This present study is to investigate changes/ effects of glutaraldehyde, microwave sterilization on Flexural strength of Auto Polymerizing (PMMA) resin.

Micro-wave Energy

Micro-waves are Radio frequency waves. Materials such as water are microwave absorbent and heat within the microwave field. Denture base acrylic resins are Transparent to microwaves. They neither absorb nor reflect microwave field nor do they heat. Metals are microwave reflectors. Important concept is that microwave heating is energy conversion.

Materials and Methods

The following materials and instruments have been used in this study.

I. Materials

- 1. **Group1**: self cure acrylic (tooth coloured) for temporization of crown and bridge in powder and liquid as supplied by *Dr.Jagdish lal sethi, Wazipur India Limited* under the brand name *SC-10*.
- 2. **Group2:** self cure acrylic repair resin denture base polymer resin in powder and liquid form as supplied by DPI under the brand name of DPI-RR cold cure.

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| Specifications: | |
|---------------------|------------|
| Power output (Max.) | - 900 watt |
| Turn Table | - Glass |
| Interior | - Acrylic |

III. **Disinfecting and sterilization solution** 2.0 % Glutartadehyde w/v (1 lt. Pack) with a separate activating powder supplied by *Johnson & Johnson* – under the brand name of *CIDEX*.

Testing Apparatus: Instron Testing Machine Model 6025. (Fig.1)

IV. Other miscellaneous materials and instruments

- 1. Rubber base impression materials (putty)
- 2. Glass measuring Jar
- 3. Glass measuring jar
- 4. Stainless steel spatula
- 5. Glass Plate
- 6. Digital weighing machine.

Preparation of the moulds:

For measuring Transverse strength:

One M-steel bar of uniform diameter of approximately 70mm length, 12mm width and 3mm of thickness was used to prepare the mould in silicon material (rubber base putty material).

Equal amounts of silicone impression materials are mixed and placed on a plastic tray and the M-steel bar placed on it and a glass slab is placed over it so that mold is formed of without distraction of the silicon material.

Preparation of Specimens for Transverse strength:

Sixty self cured specimens were made. For each group ten specimens were made. Thirty specimens of tooth colored and 30 specimens of pink colored (repair material) were made.

The monomer-poly ratio was maintained at 2:3 by volume / weight for all groups. A pipette was used to transfer the required volume of monomer to a clean dry measuring jar. The polymer was measured by digital weighing machine and transferred to the dap-an-dish containing monomer. Thorough mixing was done with straight spatula. The mix was flown into the mould while tapping to avoid any air bubbles. A glass slab was placed over the mould to remove the excess material as flash, and to get a smooth surface. By the by the weight of the glass may act as pressure agent. The mold was allowed to polymerize for 10 minutes and the specimens were

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carefully separated from the mould. Set specimens were then trimmed for any excess borders with wheel stone and five graded sand paper.

Coding of Specimens:

Specimens prepared out of tooth colored are coded as 1. Specimens prepared with pink colored repair resin (pink) are coded as 2.

With the tooth colored 30 specimens are made for testing impact strength coded as 1 Ai. 1 Bi, 1 Ci .

With the repair resin (pink colored) 30 specimens were made for testing impact strength. They are coded as 2 Ai, 2 Bi, 2 Ci, for impact.

A – Indicates control. (10 specimens each)B- Indicates micro-wave sterilized.C- Indicates chemical sterilized.

For each material, 10 specimens were used control. 10 micro-wave oven sterilization and 10 for chemical sterilization.

Control : The specimens used as control are placed in water for 10 hours. 1 Ai, 2 Ai each.

Chemical Sterilization: According to the manufacturer recommendation the 20 specimens are placed in the chemical solution for 10 hours.(**Fig.2**) 1 Ci, 2 Ci 10 specimens each.

Micro-wave sterilization:

Twenty specimens were placed in the micro wave on the turn table. For 15 minutes adjusted at 650 W power. A Borosil bowl filled with water was placed to protect the magnetron. A Borosil bowl with water placed parallel to the resin blocks to absorb heat generated to save the magnetron. Ten specimen each of 1 Bi and 2 Bi were used(**Fig.3**). There was exhaust provision also to drive out the heat generated by resin blocks during micro wave exposure. The micro wave energy was focused at an power adjusted to 650 watt. Continuously for 15 minutes.

The specimens were then removed from the oven and were ready for testing.

Testing for Flexural strength

Procedure:

A bar of rectangular cross section is tested in flexure at a beam as follows: The bar rest on two supports and is loaded by means of loading nose midway between supports(**Fig.4.**). A properly calibrated testing machine(Instron Testing Machine Model 6025) that can be

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operated at constant rates of cross-head motion over the range indicated and in which the error in the load measuring system shall not exceed ± 1 % of maximum load expected to be measured(**Fig.1.**) It shall be equipped with a deflection measuring device. The stiffness of the testing machine shall be such that the total elastic deformation of the system does not exceed 1 % of the total deflection of the test specimen during the testing, or appropriate correction shall be made.

Conditioning: Conditioning the test specimens at $23 \pm 2^{\circ}$ C and 50 ± 5 % relative humidity for not less than 40 hours prior to testing in accordance with procedure A of practice D 618 for those tests where conditioning is required ¹⁵. Procedure: Untested specimen are used for each measurement determine the support span to be used and set the support span to with in 1 % of the determined value. The cross head motion is calculated with the formula.

R=ZL²/6d

- R = Speed of movement.
 Z = Rate of stain 0.01 constant
 L = Span length (16 x thickness of specimen)
- d = thickness of the sample.

The speed of the movement for this test calculated was 0.02 mm/ min. The sample is placed on the supports and applies the load to the specimen at the specified cross head speed of 0.026 mm/min. The load deflection curves may be plotted to determine the flexural yield strengths.

S = 3 PL / 2bd²

- Where P is the load at fracture. L = Length between the jig wedges support span in (mm)
- b= width of sample tested.
- d = depth of the sample tested in (mm)

Results

The results of Flexural Strength after microwave and chemical sterilization were shown (**Table I to IV**). Their comparisons are shown (**Tables V to VII**)

The flexural strength of sterilized specimens is compared with specimens stored in water for 10 hours. To obtain any significant differences between the data obtained for the groups of material tested. A one-way analysis of variance (ANOVA) and post HOC Tests was under taken. The flexural strength of SC-10 (tooth colored) material did not change significantly (P>0.05) by chemical and micro wave sterilization.

The Flexural strengths of repair resin (pink) varied with control. There was a significant change in the flexural strength of repair resin after chemical sterilization. Significantly showing reduced result less than 0.05 (P<0.05).

| Table .1 Flexural Strength Of Tooth Colored Resin (Flexural StrengthN/mm²) | | | | | | | |
|---|------------------|---|---|--|--|--|--|
| Specimen | Control: 1 Af | After micro wave sterilization : 1Bf | After Chemical Sterilization – 1Cf | | | | |
| 1. | 61.21 | 63.52 | 67.91 | | | | |
| 2. | 63.13 | 62.67 | 57.53 | | | | |
| 3. | 61.20 | 59.69 | 61.01 | | | | |
| 4. | 63.11 | 60.83 | 59.60 | | | | |
| 5. | 66.77 | 57.53 | 53.10 | | | | |
| 6. | 55.62 | 62.68 | 53.15 | | | | |
| 7. | 56.20 | 59.68 | 59.75 | | | | |
| 8. | 66.78 | 60.80 | 63.80 | | | | |
| 9. | 56.20 | 63.50 | 61.15 | | | | |
| 10. | 55.60 | 57.52 | 61.10 | | | | |

| (Flexural StrengthN/mm ²) | | | | | | | |
|--|------------------|---|---|--|--|--|--|
| Specimen | Control: 2 Af | After micro wave sterilization : 2Bf | After Chemical Sterilization – 2Cf | | | | |
| 1. | 60.90 | 56.92 | 56.10 | | | | |
| 2. | 54.03 | 57.28 | 56.62 | | | | |
| 3. | 60.20 | 54.09 | 53.95 | | | | |
| 4. | 62.81 | 61.71 | 56.63 | | | | |
| 5. | 56.97 | 55.18 | 55.25 | | | | |
| 6. | 60.29 | 57.55 | 56.72 | | | | |
| 7. | 55.20 | 55.20 | 56.15 | | | | |
| 8. | 60.25 | 54.20 | 58.18 | | | | |
| 9. | 59.80 | 60.70 | 55.40 | | | | |
| 10. | 58.32 | 59.98 | 58.20 | | | | |

Table .2. Flexural Strength Of Repair Resin

| Table.3. Flexural Strength Of Tooth Colored Resin | | | | | | | | | |
|---|----|-------|------|---------------|-----------------------|-----------------------|-------|-------|--|
| (Mean & SD.Values By Groups) | | | | | | | | | |
| Group | N | Mean | SD | Std. Error | 95 % Confide for N | ence Interval lean | Min | Max | |
| | | | | | Lower | Upper | | | |
| | | | | | Bound | Bound | | | |
| Control (1Af) | 10 | 59.98 | 3.89 | 1.23 | 57.19 | 62.76 | 55.60 | 66.77 | |
| Micro-wave (1Bf) | 10 | 60.84 | 2.25 | 0.71 | 59.23 | 62.45 | 57.52 | 63.52 | |
| Chemical (1Cf) | 10 | 59.71 | 4.46 | 1.41 | 56.51 | 62.45 | 53.10 | 67.91 | |
| Total | 30 | 60.18 | 3.56 | 0.65 | 58.84 | 61.50 | 53.10 | 67.91 | |

| Table.4. Flexural Strength Of Repair Resin (Mean & SD.Values By Groups) | | | | | | | | |
|--|----|-------|------|-------|----------------|-----------------------------|-------|-------|
| Group | Ν | Mean | SD | Std. | 95 % Con fc | fidence Interval or Mean | Min | Max |
| | | | | | Lower Bound | Upper Bound | | |
| Control (1Af) | 10 | 58.88 | 2.73 | 0 .86 | 56.93 | 60.83 | 54.03 | 62.81 |
| Micro-wave (1Bf) | 10 | 57.28 | 2.73 | 0.86 | 55.33 | 59.54 | 54.09 | 61.71 |
| Chemical (1Cf) | 10 | 55.32 | 1.47 | 0.47 | 54.26 | 56.37 | 53.24 | 58.18 |
| Total | 30 | 57.16 | 2.74 | 0.55 | 56.14 | 58.18 | 53.24 | 62.81 |

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Fig. 1. Instron Testing Machine Model 6025



Fig.2. specimen in 2% Glutarldehyde (activated)



Fig. 3. Photograph showing specimens placed on the Turn table of oven along with the Borocil Bowl (filled with water)



Fig.4. Beam model for measuring the flexural strength

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| Table.5. ANOVA test for Tooth Colored Resin | | | | | | | | |
|---|-------------------|----|----------------|------------|-------|--|--|--|
| Parameter | Sum of Squares | Df | Mean Square | F value | Sig. | | | |
| Between Groups | 6.983 | 2 | 3.492 | 0.261 | 0.772 | | | |
| Within Groups | 361.252 | 27 | 13.380 | | | | | |
| Total | 368.235 | 29 | | | | | | |

| Table.6. ANOVA test for Repair Resin | | | | | | | |
|--------------------------------------|-------------------|----|----------------|------------|-------|--|--|
| Parameter | Sum of Squares | Df | Mean Square | F value | Sig. | | |
| Between Groups | 63.773 | 2 | 31.887 | 0.261 | 0.772 | | |
| Within Groups | 153.835 | 27 | 5.698 | 5.59 | .009 | | |
| Total | 217.608 | 29 | | | | | |

Discussion

Sterilization is compulsory for any provisional restoration whether removable or fixed provisional restoration. Whether we do chemical sterilization or micro wave sterilization aim is that sterilization technique should not effect the properties of the restoration. The aim of this study is to determine any effects of sterilization on strengths of the two resin used for temporary bridge work and repair resin after chemical and micro wave sterilization. Till 1985 chemical sterilization was only method for Acrylics, Rohrer and Bulard ¹³ 1985 reported micro wave energy could be used for consistent sterilization and that aerobic spore forming micro organisms. The most resistant to sterilization could be eliminated with in a 15 mints. Micro wave energy exposure. They also reported acrylic denture subjected to micro wave energy were dimensionally identical before and after exposure. According to ADA recommendation the sterilization can be achieved with chemicals in 10 hrs. -12 hrs. Rohrer and Bulard ¹³ reported sterilization in 15 min. with micro wave exposure. Tooth colored acrylic resin is the material of choice for provisional coverage of teeth that have been prepared for fixed prosthodontic restorations ¹¹

Flexural Strength ¹⁸. Flexural strength, transverse strength or modulus of rupture, is essentially a strength test of beam supported at each end under a static load. The study evaluated the effects of chemical (Glutaraldehyde) and micro wave sterilization on flexural and impact strengths of autopolymerising resins used for denture repairs and in fabrications of temporary Crown and bridge restorations. Samples were prepared according to ASTM standards. For

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chemical sterilization samples were kept in a vessel containing 2 % Glutaraldehyde commercially available as CIDEX along with activator. Once activated remains effective till 14 days. The specimens were kept for 10 hrs. As recommended by ADA. For micro wave sterilization, the specimens were kept on the turn table of micro wave oven adjusted to 500 w (net output) for 15 min. along with a Borosil vessel filled with water. According to recommendation of Rohrer and bulard ¹⁹. For control specimens were kept in water for 10 min. before testing for strengths. Flexural strengths of all the groups were tested by Instron machine. The result of the tests indicates that Flexural strength of Tooth Colored resin (1 Bf) not affected by the micro sterilization, when compared with the control group 1 Af the mean transverse strength were 60.84 N/mm2 and 59.98 N/mm2 and chemical sterilization were decreased by 59.71 N/mm2. It is clear that tooth colored resin was not much effected by chemical sterilization and was little bit increased by micro wave sterilization. This increase in transverse strength may be attributed to the further polymerization of residual monomer.

Flexural strength of repair resin after chemical sterilization affected significantly the mean Flexural strength of repair resin after chemical sterilization (2 Cf) was 55.31 N/mm2 when compared with control 58.88 N/m,m2 and micro wave sterilization coincides with the shen, Javid, and Frank's ¹⁰ 1989 investigation on the effect of glutaraldehyde base disinfectants on denture resins who reported that Flexural strength was affected by the length of immersion time when repair resin was immersed in Glutaraldehyde the Flexural strength was decreased. Certain components of disinfectants may have penetrated in to the material and resulted in reduction of flexural strength. Both the sterilization methods can be safely applied in everyday practice for the sterilization of Auto polymerizing resins. The micro wave method seems to be a reliable alternative having the advantage of less time consumption.

CONCLUSION

Under the conditions of this study, the following conclusions may be made.

- 1. The Flexural Strength of tooth colored material (SC-10) did not change significantly by either chemical or micro wave sterilization.
- 2. The Flexural strength of repair resin varied with control groups There was significant reduction in strength after chemical sterilization for 10 hours.
- 3. By micro wave sterilization there was no significant change in Flexural Strength of both resins (Tooth Colored and Repair Resin).
- 4. It is concluded that micro wave sterilization can be a better method because of less time consumption and no/less adverse effects when compared to chemical (Glutaraldehyde) sterilization.

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| Table .7.Post Hoc Tests Multiple Comparisons Dependent Variable | | | | | | | | |
|---|-----------|-----------------------------|-----------|-------|--|--|--|--|
| Group(I) | Group(J) | Mean Difference (I-J) | Std.Error | Sig. | | | | |
| Control | Microwave | 1.5980 | 1.0675 | .146 | | | | |
| | Chemical | 3.5650 | 1.0675 | .002 | | | | |
| Microwave | Control | - 1.5980 | 1.0675 | .146 | | | | |
| | Chemical | 1.9670 | 1.0675 | 0.76 | | | | |
| Chemical | Control | -3.5650 | 1.0675 | 0.002 | | | | |
| | Microwave | -1.9670 | 1.0675 | 0.76 | | | | |

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