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COMPARISON OF RESIDUAL MONOMER AND WATER ABSORPTION IN ACRYLIC RESIN SAMPLES PROCESSED WITH MICROWAVE AND CONVENTIONAL HEAT CURE POLYMERIZATION METHODS – INVITRO STUDY.

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ABSTRACT:

The different denture base materials and their processing techniques are developing in the field of removable and fixed prosthodontics. The polymers, especially acrylic resins are entered in this field more than 70 years ago and it is most widely used and material of choice around 80% world wide. Initially acrylic resins were polymerized by heat, later they developed using chemical accelerators for polymerization and termed as self-cure resins. The use of microwave energy to polymerize acrylic denture base in a 400 watt microwave oven for 2.5 minutes was reported in 1968 and properties ware discussed. The present study is designed to compare the residual monomer content in 24,48,72,96 and 120 hours periods of time and water absorption after 10 days in acrylic resin samples processed with conventional heat cure polymerization method and microwave polymerization method.

KEYWORDS: **Acrylic resins, Microwave processing, Residual monomer, Water absorption.**

INTRODUCTION

Acrylic resins have been used for denture fabrication over six decades. The most popular denture base resin material is heat cured poly (methyl methacrylate) due to its many advantages such as dimensional stability, handling characteristics, ease of processing, capacity to mold in complex forms with application of pressure and heat. All the dentures are constructed from poly (methyl methacrylate) material by using conventional polymer: monomer dough molding process and cured using a water bath system. Other than heat treatment poly (methyl methacrylate) resin also may be polymerized using microwave energy. This technique requires a specially formulated non- metallic flask (MUFLA- VIPI-STG) and conventional microwave oven which is used to generate the thermal energy required for polymerization.[1] The major advantage of this technique is the speed with which polymerization accomplished, less energy and more economical.[2]

Despite the above mentioned methods to polymerize denture base materials, the conversion of monomers to polymers is never complete and some unreacted monomer called residual monomers are left in the denture base polymers.[3] The amount of residual monomer content in conventional heat cured acrylic resin is 0.2 to 0.5% and in microwave cured acrylic resins it is 1.4% and in self cured resins it is about 5% .[4] Poly methyl methacrylate absorbs relatively small amount (0.69 mg/cm^2) of water when placed in aqueous environment like in mouth, in solutions of denture cleansers and water. The absorption of water molecules within the polymerized mass causes slight expansion and interferes with the polymerized chains, there by acts as plasticizers and leads to alterations in physical and mechanical properties.[5] Considering all the above mentioned factors, this invitro study was conducted to compare the residual monomer content in 24,48,72,96 and 120 hours periods of time and water absorption after 10 days in acrylic resin samples processed with conventional heat cure polymerization method and microwave polymerization method.

Materials and Methods

Preparation of standard mould This invitro study was conducted to evaluate and compare the amount of residual monomer in 24,48,72,96 and 120 hrs periods of time and water absorption in acrylic resin samples (10×10×3 mm) processed with microwave polymerization method and with conventional heat cure polymerization method. The design of Standardized mold was illustrated in (**Fig.1)** which is made from carbon steel metal for the purpose of fabrication of putty indices and it consists of a base (43×43 mm) with guiding pins, middle compartment

consist mold spaces with guiding holes and covering lid with guiding holes. The mold spaces in middle compartment were milled precisely to produce four (10×10×3mm) square shaped spaces which are equidistant from each other. This mold was designed in such a way that it can open on both sides for easy removal of the putty indices.

Acrylic specimens preparations Putty (Exaflex, GC America Inc) indices (120 numbers) prepared from the mold and 60 numbers invested with dental stone type III in Brass metal flask (Varsity Flask Jabber Company No: 7) for heat cure polymerization and 60 numbers in non metallic flask for microwave polymerization. After the dental stone has completely set, the two portions of both brass flask and non metallic flask opened carefully. Putty indices were recovered from the lower portion. Mold separator was carefully applied to all mold spaces for both brass and non metallic flasks. Heat cure material (DPI –H) was mixed in proportion of 30: 10 ml by volume in the porcelain mixing jar. During dough stage the material was packed and trial closure performed to eliminate excess flash. Acrylic resin samples were cured in (Delta-Poly Bath) at 74°c for 2 hr and terminal curing done at 100°c for1 hr and then bench cooled. Acrylic specimens were recovered and excess resin flash was removed with fine abrasive without damaging specimen surfaces and dimensions. Nonmetallic flask (Mufla) was placed in the microwave oven (Samsung- Mw – 73v) and cured at 450 watts for 2 min and then the flask was turned upside down and again cured at 450 watts for 2 min and flask was allowed to cool to the room temperature for 1 hr.

Residual monomer evaluation Residual monomer content was determined by using a U V Double beam spectrophotometer (2201 Systronics) (**Fig.2).** When light pass through a liquid substance, some of light gets absorbed and by measuring this with Double Beam Spectrometer the concentration of liquid substance can be analyzed. The amount of absorption was depending on the concentration of that substance. A stock solution of 1 % v/v methyl methacrylate was prepared by dissolving 1 ml of methyl methacrylate in 99 ml of distilled water. From the above stock solution a series of concentrations are prepared between 0.005 % to 0.8% by diluting with distilled water. By using Double Beam Spectroscopy at 210 nm the absorbencies of these standard solutions were determined and a standard graph was plotted between concentration and absorbance (**Fig.3).** Each specimen immediately after preparation, placed in screw capped containers consists of 20 ml of distilled water and stored at room temperature for 24,48,72,96 and 120 hrs. The solutions were transferred and they were subjected to U V Double beam spectroscopy at 210 nm. The unknown amount of residual monomer leached into the distilled water was analyzed and values were compared with standard graph.

Water absorption The samples which were prepared to evaluate water absorption were weighed before water

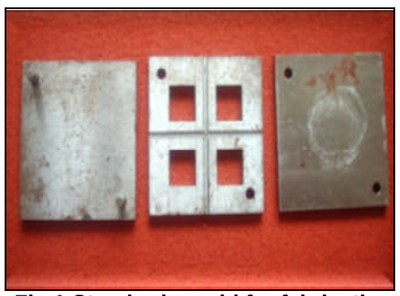


Fig.1 Standard mould for fabrication of heat cure acrylic samples.

Fig.2 UVDouble beam spectrometer

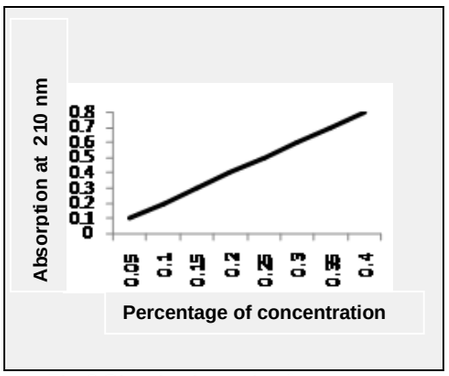


Fig.3.Standard graph of monomer Concentration

Fig.4.Desiccator

immersion (dry weight) with digital balancer (CITIZEN) with 0.001gm accuracy. These samples were kept in screw capped containers consisting of 5ml of distilled water at room temperature for 10 days. After 10 days each sample was recovered, surface droplets were removed with tissue paper and weighed (wet weight). The acrylic resin samples were then placed in a Desiccator **(Fig.4)** consisting of copper sulphate until it gets the original dry weight (±0.001gm) i.e. desiccated weight. The percentage of water absorption was calculated by subtracting desiccated weight from wet weight.

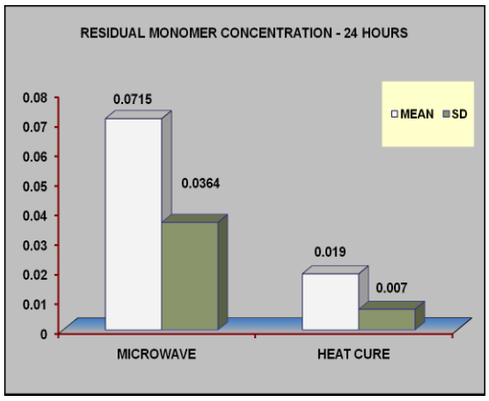
Results

Mean residual monomer measurement and standard deviations of the 2 groups are made and listed (**Table I** and **Graph-I)** . The mean values in between the two processing methods shows *'p'* value is highly statistically significant. This means that, the residual monomer in microwave processing method is having higher concentration than heat cure processing method. Within different time intervals, heat cure processed samples showed residual monomer concentration in first 24, 48 and 72 hrs and then these values remain constant after 72, 96 and 120hrs. Where as in microwave processed samples has shown maximum increase in residual monomer concentration in first 24,48,72 and 96 hrs and then remains constant after 96 and 120 hrs.

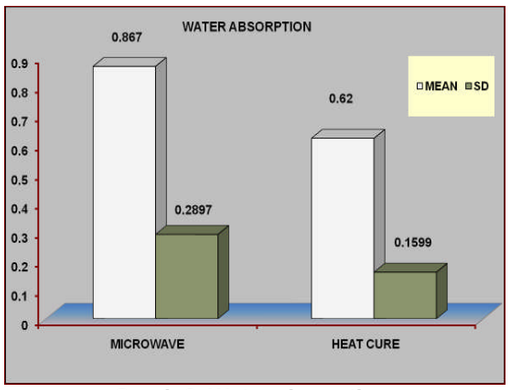
The mean values in water absorption of the two variables i.e. microwave (0.867) and heat curing (0.62) processing methods are shown (**Table II and Graph-II).** The *'p'* value (0.0298) shows that it is statistically significant and the amount of water absorption of microwave acrylic resin samples was higher than the heat cure acrylic resin samples.

Statistical analysis

The measured values are subjected to statistical analysis by using "unpaired *t* test" to know any significant difference between the two variables. The 'mean', 'standard deviation' and '*p*' values are calculated for the variables. In this present study $p < 0.05$ is considered as the level of significance. The results of this in vitro study are tabulated as follows.



Graph.1. Residual monomer concentration after 24 hours



Graph.2. Water absorption.

Discussion

In spite of many improvements in the material science and techniques, the conversion of monomer to polymer is never complete and some unreacted monomers called residual monomers are left in the denture base polymers[3]. This left out residual monomer has proved to be cytotoxic causing erythema, erosion of mucous membrane, burning sensation of tongue and oral mucosa[6]. This residual monomer in the polymerized acrylic resins depends upon the chemical composition, the polymer to monomer ratio, the manipulative variables and storage in water. Polymerization with microwave energy was introduced as early as in 1968 by Nischii[7]. In 1984 the fiber reinforced dental flasks are substituted with the brass flasks and water bath is replaced by microwave oven for microwave polymerization method. The uniform heating on either side of the flask and rapid raise of temperature with the resultant shortage of polymerization time makes microwave curing methods superior to the conventional water bath curing methods.

The microwave heating is independent of thermal conductivity, so it leads to a curing cycle without the production of any unwanted exothermic heat[7]. In this present study the residual monomer content was high (0.0715) in microwave polymerized specimens when compared to heat cure specimens (0.019), the reason could be in microwave curing method, the monomer molecules vibrate by being exposed to a high frequency electromagnetic field. This phenomenon leads to intermolecular collisions that generate the heat for activation process and because of high power applied to the system, the benzoyl peroxide decomposition and

Table.1 Comparison of mean and standard deviation values of residual monomer analysis between microwave and heat cure processing techniques.

Sl.No.	TIME (hr)	MICROWAVE		HEATCURE		'P' VALUE
		MEAN	SD	MEAN	SD	
1	24	0.0715	0.0364	0.019	O.0070	0.0003
2	48	0.14	0.014	0.028	0.009	0.0001
3	72	0.16	0.033	0.032	0.011	0.0001
4	96	0.188	0.036	0.032	0.007	0.0001
5	120	0.188	0.022	0.037	0.010	0.0001

Table.2 Comparison of mean and standard deviation of water absorption after 10 days of storage between microwave and heat cure processing techniques.

Sl.No.	POLYMERIZATION METHOD	MEAN	SD	'P '- VALUE
1	MICROWAVE	0.867	0.289	0.0298
2	HEAT CURE	0.62	0.159	

polymerization reaction becomes faster. A rapid polymer structure hinders the conversion of MMA monomers especially at curing temperatures lower than the glass transition temperature (Tg) of polymer. Where as the residual monomer concentration in heat cured denture base polymers was considerably lower than microwave polymers it was because of the higher the curing temperature, which can be as high as glass transition temperature (Tg) of the matrix phase of heat cured denture base polymer (97 to 100°c). Above the Tg of the polymer the monomers of the resins have a better ability to polymerize due to higher molecular chain motions and neutralization of the immobilization of methyl methacrylate in the glassy polymer at higher temperature.[7,8,9]

In this present study within different time intervals, heat cure processed samples showed maximum increase of residual monomer concentration in first 24, 48 and 72 hrs and then these values remain constant after 96 and 120hrs.Where as in microwave processed samples has shown increase in residual monomer concentration in first 24, 48, 72 and 96 hrs and then remains constant after 120 hrs. The decrease in the daily release of monomer occurred as a result of the monomer diffusion in water and by continuous polymerization promoted by the active radicals found in the polymer chains. After 48 hrs the content of monomer release was very low. Due to the cytotoxic effect of residual monomer, it is suggested that the prosthesis should be soaked in water for at least 48 hrs before denture insertion and the results are coinciding with investigating conducted by Daniela Maffei Botega et al[10]. In the present study the amount of water absorption was higher for microwave polymerized specimens (0.867) than heat cured specimens (0.62). This is because, when the level of residual monomer is high, the number of voids in the resin after leaching of residual monomer will be high and water intake will also be high.

CONCLUSION

Within the limitations of this study, the following conclusions are drawn.

1. The results of residual monomer content analysis of acrylic resin samples processed through conventional heat cure and microwave curing methods at 24,48,72,96 and 120 hrs period of time intervals has shown that there was a high statistically significant difference between these two processing methods. The residual monomer content of microwave specimens has shown higher values than heat cure specimens for the above said periods of time.
2. The residual monomer analysis at different time intervals has shown that the amount of leached residual monomer into the distilled water was higher in first 48 hrs (0.14 for microwave & 0.028 for heat cure resin samples) water storage and later it was greatly decreased.
3. The water absorption of acrylic resin samples has shown a statistically significant difference between these two processing methods (p = 0.029). The amount of water absorption of microwave acrylic resin samples was higher than heat cure acrylic resin specimens.

References

1. S.G. I lbay and S.Guvener. Processing dentures using a microwave technique. Journal of Oral Rehabilitation 1994; Volume 21:103-109. http://dx.doi.org/10.1111/j.1365-2842.1994.tb01129.x

2. Robert L. Schneider, DDS, MS, Erik R. Curtis, DDS, and James M.S. Clanct, DDS, MSc. Tensile bond strength of acrylic resin denture teeth to a microwave or heat processed denture base. J Prosthet Dent 2002; 88:145-150.
3. J. A. Bartoloni, D. F. Murchison, D. T. Wofford & N. K. Sarkar. Degree of conversion in denture base materials for varied Polymerization techniques. Journal of Oral Rehabilitation2000; 27: 488–493.
4. M. Fletcher. S Purnaveja. W. M Amin, G.M. Ritche, S Morsdians and A. W. Dodo. The level of residual monomer in self curing denture base materials .J Dent Res 1983; 62:118 – 120.
5. Kenneth J. Anusavice, PHD, DMD. Phillip's Science of Dental Materials. 11th edi.
6. Janaina Habib Jorge, DDS, Eunice Teresinha Giampaolo, DDS, PhD, Ana Lucia Machado, DDS, PhD and Carlos Eduardo Vergani, DDS, PhD .Cytotoxicity of denture base acrylic resins. A literature review. J Prosthet Dent 2003; 90:190-3.
7. C.-P. Laia, M.-H. Tsaia, M. Chena, H.-S. Changb, H.-H. Tayb .Morphology and properties of denture acrylic resins cured by microwave energy and conventional water bath. Dental Materials 2004; 20: 133–141.
8. Dogan, B. Bek, N. N. Cevikf- and A. Usanmazt. The effect of preparation conditions of acrylic denture base materials on the level of residual monomer, mechanical properties and water absorption .Journal of Dentistry1995;23(5):313-318 . http://dx.doi.org/10.1016/0300-5712(94)00002-W
9. Pekka K. Vallittu, I. Eystein Ruyter, Sebnem Buykuilmaz. Effect of polymerization temperature and time on the residual monomer content of denture base polymers. Eur J Oral Sci 1998; 106: 588–593.
10. Botega DM, Machado TS, Nunes de Mello JA, Rodrigues Garcia RCM, Del Bel Cury AA. Polymerization time for a microwave-cured acrylic resin with multiple flasks. Braz Oral Res 2004; 18(1):23-8.http://dx.doi.org/10.1590/S1806-83242004000100005

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