Thermal curves of acrylic resins in microwave curing

Curvas térmicas de resinas acrílicas polimerizadas por energia de microondas

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Abstract

The use of microwave polymerization on acrylic resins is a clean and easy method to make complete dentures. It is observed that this kind of process may cause a higher porosity when compared to the conventional heated water bath, probably because of the excessive temperature increase. The objective of this study was the qualitative observation of the thermal curves generated on acrylic by the application of microwave energy in different polymerization cycles. Wax model dentures were reproduced, resulting in four specimens, within each one 7 thermocouples were positioned, having different reading sites of the specimens while the resin was polymerized in a conventional microwave oven. Four cycles were used: 1) three minutes at 475W; 2) 13 minutes at 95W + 1,5 minute at 475W; 3) three minutes at 475W with 150ml of water; and 4) seven minutes at 95W + seven minutes at 95W. The thermocouples were linked to data acquisition equipment, resulting in time-versus-temperature charts. It was concluded that lower power settings led to lower temperature increases. The use of minimum water load caused lower temperature peaks. The used method has a great value for temperature readings when polymerizing microwave acrylic resins.

Uniterms

Acrylic resins; microwaves; light; porosity; surface properties; dental equipment

Introduction

The use of acrylic resins in Dentistry involves a large range of applications. Therefore, the improvement of its physical and mechanical properties becomes necessary. Particularly, the thermo-activated, polymethylmethacrylate-based acrylic resin has become the main choice material for total or partial denture bases confection [2], since it is dimensionally stable, has low citotoxicity[16] and is relatively cheap.

In the past few years, the microwave activation has been widely studied and applied in dental practice, for it is technically easy, takes lower processing time[4,6] and has similar properties to the acrylic resins processed in heated water bath, concerning dimensional stability[1,8,12,14-5,19], low residual monomer leaching[11], satisfactory color stability[7], hardness[7,9,10,18] , and transversal resistance[10,17-8], even in cases of rebasing or repairing[13,21].

Although there are some advantages facing heated water bath, it was observed that the porosity number, after microwave polymerization, was significantly higher in thick specimens or around metallic structures[18,20], which can decrease the prosthetic quality. An explanation to this would be that, different than the water bath, where the temperature applied over the monomer tends to be similar to the environment one, it is difficult to evaluate how much energy must be given to the resin mass without causing monomer ebullition, which consequently causes porosity in the final product. To avoid this inconvenience, some studies[3,17] suggest changes in the working method or in the composition of the resins, so the polymerization process can run in microwave ovens.

Another probable solution to this problem could be the use of an ideal cycle that keeps the monomer temperature from reaching its boiling point. However, the solution was not found in literature, since the consulted studies use various different cycles, ranging from the ones indicated by resin manufacturers to the ones suggested by other authors[4,17]. Another reason that makes cycle standardization difficult is the difference among microwave ovens and their specifications, which makes people work with approximated values.

Because of that, the purpose of this study is, by observing the thermal curves generated by the application of microwave energy in different cycles on the acrylic resin, to qualitatively evaluate the power-versus-time combinations and minimum loads, taking the boiling point of the monomer as a parameter, in order to produce a behavioral analysis of the material in different cycles, searching for basis for the development of an ideal polymerization cycle, or one with optimized results. To achieve this objective, a new evaluation methodology will be introduced in literature, wherefrom it will be possible to evaluate the temperature in many regions of the resin mass in real time.

Material And Methods

Specimens Manufacturing

From a master cast correspondent to a totally edentulous maxilla, a master impression of laboratorial silicone was obtained, over which type III plaster casts were reproduced. From one plaster cast, a wax model denture was made on the dental foundation area, with a palate covering of uniform thickness (2mm) obtained through the double thickness of the wax lamina. It was established: occlusal width of the wax model denture, 12mm; the distance between the bottom of the labial vestibule and the edge of the model denture in the anterior region, 15mm; and the distance between the tuber and the edge of the model denture in the posterior region, 5mm. Two wax sprues formers were positioned on the posterior region of the model denture. This set was duplicated with laboratorial silicone, obtaining a matrix for the standardization of the trial denture. Thus, the plaster casts were incased on the silicone matrix and liquefied wax was poured inside it all the way to the top, through the sprues. After cooling the set was removed, thus achieving precise copies. This procedure was repeated four times.

Positioning of thermocouples and inclusion in flask:

Seven type T (chromel-alumel) thermocouples (30AWG x 2m, TT-T-30 Omega, Omega Engineering Ltd., Stamford, Connecticut, USA) were prepared for each specimens; 3mm of the extremity of each thermocouple were prepared and welded. These tips were introduced in the wax model denture in different measurement sites, as indicated in Figure 1. Sites 1, 3 and 5 are related to the external denture surface and sites 2, 4 and 6, to the interior of the resin mass. Site 7 corresponds to the posterior palate region.

The set was invested in fiber-reinforced plastic flasks, specific for microwave oven usage (GC Corporation, Tokyo, Japan), in order to the thermocouple extremity – opposite to the sensor – remain free and out of the flask, allowing connection to the reading system. The wax elimination was performed via heated water bath. The acrylic resin (Acron-MC, GC Corporation,

FIGURE 1 – Measurement sites in specimen

Tokyo, Japan) was mixed according manufacturer's instructions and placed into the mold (30 cc of powder and 9ml of liquid). This way, the active extremities of the thermocouples, once inserted in wax, were now inserted in the same position in the resin mass to be polymerized. Four specimens were made through this method, and each one of them was put through one of the polymerization processes described below.

Four polymerization cycles took place, as Table 1 indicates. The first two ones are recommended by resin manufacturers, the first one belonging to Acron-MC (3 minutes/475 W) and the second one to Justi (13 minutes/95 W followed by 1.5 minute/475 W). Cycle three is a variation of the Acron-MC cycle, but with a minimum water load of 150ml close to the flask, inside the microwave oven3. Cycle four was adapted from literature[17], with a seven minutes/95 W x seven minutes/95 W application, horizontally inverting the position of the flask between the two irradiation periods. Some adaptations in the cycle powers were made, for microwave ovens used in literature and the one in our experiment are different regarding models and nominal powers, thus demanding value roundoffs.

Reading system and data collecting:

The data collecting system consists on a set of equipment (Figure 2), starting at the microwave oven itself (Brastemp DES 950W/12,7A, Multibras Eletrodomesticos S.A., Sao Paulo, Brazil). Inside the oven, the specimens were always positioned with the anterior region of the model denture facing the magnetron, and in the same position of the tray. The seven thermocouples passed underneath the door of the oven towards the external environment – a physical condition that disables the rotatory plate to rotate during the experiment – to be connected to the Switch Control Unit 3488A (Hewlett-

Table 1 – Polymerization cycles in microwave oven

CYCLE 1	3 min/475W
CYCLE 2	13 min/95W + 1.5 min/475W
CYCLE 3	3 min/475W with 150ml of water
CYCLE 4	7 min/95W x 7 min/95W

min = minutes
W = watt

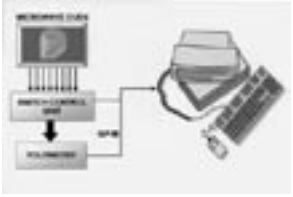

FIGURE 2 – Data collecting system

Packard, Palo Alto, California, USA). In this unit, each thermocouple was connected to an input channel. The purpose of this device is to read the seven channels in a sequential and individual order. Hence, the Switch Control Unit cycle lasted seven seconds for seven thermocouple channels, thus a thermocouple being read every second. This way, there is only one channel with sequential reading as an output for a precision digital voltmeter (7081, Solartron Instruments, Farnborough, Hampshire, England). In this device, it was registered the potential difference in Volts (V), then sent to a computer loaded with specific software for data acquisition (Global 2.33 for Windows), and tabulating potential-versus-time. An interface (GPiB) was used to coordinate system events, establishing the connections among the equipment and synchronizing all events. Using conversion tables, it was possible to convert the potential differences into temperatures and plot the resulting charts of the experiment.

Results

The obtained data are presented in charts (Figures 3 to 6), showing the temperatures of monitored sites inside the flask in function of time.

In Figure 3, it can be observed temperature peaks between 84 and 156 ºC. A great temperature increase was noticed in the measurement sites as soon as the microwave oven was turned on. Figure 4 shows that the temperature inside the flask did not trespass 80.6 ºC, while 95 W were given to the specimens. When the power setting was changed to 475 W, the temperature reached peaks of 143.5 ºC. In Figure 5, cycle three had the same power settings as cycle one, only water being the difference, and showed lower temperatures than cycle 1, with peaks of 111.8 ºC. In Figure 6, it can be noticed that cycle four presented the softest ascension curve, with isolated peaks at the end of the cycle, although not trespassing 100 ºC.

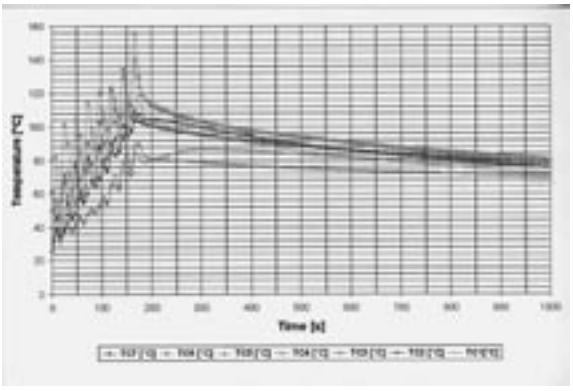

FIGURE 3 – Cycle 1: Graphic presentation of the microwave acrylic resin polymerization in function of time (3 minutes at 475 W)

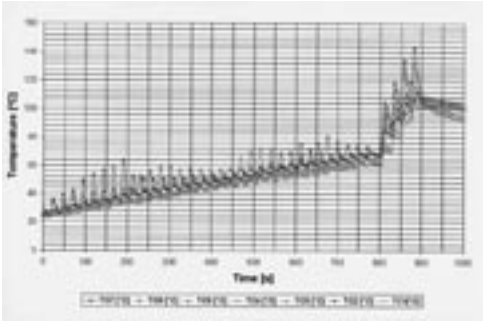

FIGURE 4 – Cycle 2: Graphic presentation of the microwave acrylic resin polymerization in function of time (13 minutes at 95 W and 1 minute at 475 W)

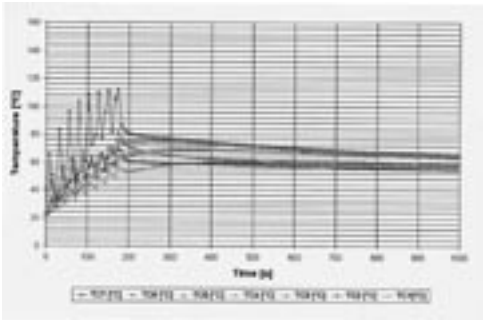

FIGURE 5 – Cycle 3: Graphic presentation of the microwave acrylic resin polymerization in function of time (3 minutes at 475 W with 150 ml of water)

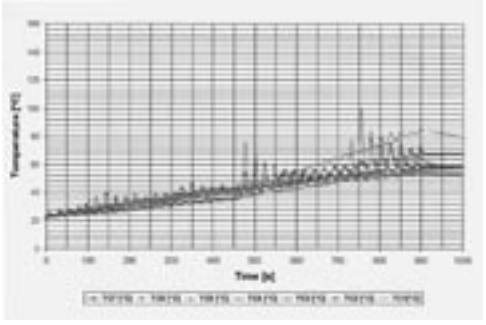

FIGURE 6 – Cycle 4: Graphic presentation of the microwave acrylic resin polymerization in function of time (7 x 7 minutes at 95 W)

Discussion

It is well known that high temperatures during the polymerization cycles of acrylic resins can induce porosity formation[10,18,20]. The monitoring of these temperatures using the conventional heated water bath cycle is not a big problem, and the amount of energy given to the resin is controlled as well. In the case of polymerization through microwaves, there is no thermal balance between the external environment and the flask. What happens is, electromagnetic energy is given to the resin; if it is incompatible to its mass, the process could end up in an excessive amount of energy.

For this reason, the monitoring of temperatures during microwave resin polymerization turns out to be an interesting method to analyze the behavior of the material, regarding the power-versus-time curve, which defines the amount of energy given by the microwave generator (magnetron). A temperature reading inside the microwave oven was made[5] during the polymerization cycle. It was suggested that it should be done with glass or carbon-fiber shielded thermocouples, in order to avoid the magnetic field generated by the magnetron to change the study results. Even so, due to the unavailability of having this kind of equipment, the present study exhibits valuable results, when analyzing different microwave resin polymerization cycles, for it allows a qualitative analysis of these cycles, besides introducing the use of a scanner that can read more than one site. It is important to emphasize that the error margin of the thermocouples used in this study is relatively small, as verified in a previous study made at the National Aerospace Research Institute – INPE (Ricardo Suterio, MS, oral communication, February 28, 2001). In this study, the temperature monitoring of the magnetron was made at constant reading, as it was also made in our study, or by interrupting cycles and making the direct measurements. The results show differences of about 3ºC among the used methods, which does not represent significant changes for our purposes.

Concerning our experiments, cycle four presented the lowest temperature increase. The possible explanation for this could be the lower microwave oven power setting (10%), applied during the entire cycle, which takes the resin to a gradual temperature increase without elevated peaks, particularly when considering that the boiling point of methylmethacrylate is 100.8ºC[2]. The same efficiency can be observed in cycle two, wherein the period prior to the power raising to 50% the temperature also had a gradual increase and without high peaks. In cycle three, where the power setting was high and time was short, the obtained temperatures were lower compared to cycle one. It happened because of the minimum load of water, where the power generated by the magnetron is distributed throughout the resin and the water, decreasing the electromagnetic energy excess over the resin[4]. In this cycle, even there being a temperature peak of 111.8ºC, it occurred at only one of the reading sites (thermocouple 7), not reproducing the behavior of the whole resin mass.

A point to be analyzed is the temperature difference between the measured sites. It was observed that in symmetric sites there were differences among the temperature peaks, which can be explained by the impossibility of the rotatory plate to work, blocking the uniform energy distribution over the specimens. It is known that electromagnetic energy is not equally distributed inside the microwave oven and, even having the flask position as a standard, the flask would be subjected to small differences among the sites, though not influencing the behavioral analysis of the material.

It is important to elicit that, before evaluating the trespassed temperature when polymerizing in a microwave oven, the exact boiling point of different resin monomers must be known beforehand, since almost none of them is constituted of methylmethacrylate only. Studies affirm that resins especially developed for microwave use show lower porosity number than conventional resins developed for heated water bath processes, when processed with a microwave oven[9]. This fact probably happens due to the addition of cross-linked agents and/or other components to the special resins, which could increase the boiling point of the monomer and thus decrease the porosity incidence.

The present study shows limitations at a quantitative analysis of generated temperatures, but attends the purpose of qualitatively comparing different polymerization cycles found in literature, and also presents a new method for temperature monitoring that in future studies will make more precise evaluations possible.

According to the experimental conditions of this study, it was possible to conclude that longer cycles with lower power settings resulted in lower temperature peaks of resin mass, i.e. cycle four, the one that presented the better qualitative performance in microwave processing in relation to the generated temperature. It is also concluded that the minimum load of water decreased the temperature peaks in cycle three significantly.

Resumo

A utilização da polimerização de resinas acrílicas por energia de microondas é um método limpo e fácil de se confeccionar próteses totais. Entretanto, é observado que este tipo de processo pode causar maior porosidade quando comparado com o banho de água aquecida convencional, provavelmente devido ao excessivo aumento de temperatura. O objetivo deste estudo foi a observação qualitativa das curvas térmicas geradas no acrílico pela aplicação de energia de microondas em diferentes ciclos de polimerização. Roletes em cera foram reproduzidos, resultando em quatro corpos-de-prova, em cada um deles foram posicionados sete termopares, obtendo diferentes sítios de leitura enquanto a resina foi polimerizada em forno de microondas convencional. Quatro ciclos foram utilizados: 1) três minutos a 475W; 2) 13 minutos a 95W + 1,5 minuto a 475W; 3) três minutos a 475W com 150ml de água; e 4) sete minutos a 95W. Os termopares foram conectados a um equipamento de aquisição de dados, resultando em gráficos de tempo-versus-temperatura. Concluiu-se que menores ajustes de potência conduziram a menores aumentos de temperatura. A utilização de carga mínima de água causou menores picos de temperatura. O método usado tem um grande valor no monitoramento de temperatura durante a polimerização de resinas acrílicas por energia de microondas.

Unitermos

Resinas acrílicas; luz; porosidade; propriedades de superfíce; equipamento odontológico.

References

1. Al-Hanbali E, Kelleway JP, Howlett JA. Acrylic denture distortion following double processing with microwaves or heat. J Dent 1991;19:176-80.
2. Anusavice KJ. Phillips' science of dental materials. 10th ed. Philadelphia: WB Saunders; 1996. p. 237-71.
3. Bafile M, Graser GN, Myers ML, Li EKH. Porosity of denture resin cured by microwave energy. J Prosthet Dent 1991;66:269-74.
4. De Clerk JP. Microwave polymerization of acrylic resins used in dental prostheses. J Prosthet Dent 1987;57:650-8.
5. Hogan PF, Mori T. Development of a method of continuous temperature measurement for microwave denture processing. Dent Mater J 1990;9:1-11.
6. Kimura H, Teraoka F, Ohnishi, Saito T, Yato M. Applications of microwave for dental technique (part 1) – dough-forming and polymerization of acrylic resins. J Osaka Univ Dent Sch 1983;23:43-9.
7. May KB, Shotwell JR, Koran A, Wang RF. Color stability: denture base resins processed with the microwave method. J Prosthet Dent 1996;76:581-9.
8. Nelson MW, Kotwal KR, Sevedge SR. Changes in vertical dimension of occlusion in conventional and microwave processing of complete dentures. J Prosthet Dent 1991;65:306-8.
9. Polyzois GL, Zissis AJ. Comparision of the toughness of denture bases cured by microwaves or dry heat. Compendium 1995;16:228-32.
10. Reitz PV, Sanders JL, Levin B. The polymerization of denture acrylic resins by microwave energy – physical properties. Quintessence Int 1985;8:547-51.
11. Sadamori S, Ganefiyanti T, Hamada T, Arima T. Influence of thickness and location on the residual monomer content of denture base cured by three processing methods. J Prosthet Dent 1994;72:19-22.
12. Sadamori S, Ishii T, Hamada T. Influence of thickness on the linear dimensional change, warpage, and water uptake of a denture base resin. Int J Prosthodont 1997;10:35-43.
13. Sadamori S, Siswomihardjo W, Kameda K, Saito A, Hamada T. Dimensional changes of relined denture bases with heat-cured, microwave activated, autopolymerizing, and visible light-cured resins – a laboratory study. Austr Dent J 1995;40:322-6.
14. Salim S, Sadamori S, Hamada T. The dimensional accuracy of rectangular acrylic resin specimens cured by three denture base processing methods. J Prosthet Dent 1992;67:879-81.
15. Sanders JL, Levin B, Reitz P. Comparision of the adaptation of acrylic resin cured by microwave energy and conventional water bath. Quintessence Int 1991;22:181-6.
16. Sheridan PJ, Koka S, Ewoldsen NO, Lefebvre CA, Lavin MT. Cytoxicity of denture base resins. Int J Prosthodont 1997;10:73-7.
17. Taubert TR, Nowlin TP. Controlling porosity in microwave processed acrylic. Trends Tech Contemp Dent Lab 1992;9:45-8.
18. Truong VT, Thomasz FGV. Comparision of denture acrylic resins cured by boiling water and microwave energy. Aust Dent J 1988;33:201-4.
19. Wallace PW, Graser GN, Myers ML, Proskin HM. Dimensional accuracy of denture resin cured by microwave energy. J Prosthet Dent 1991;66:403-9.
20. Yamauchi M, Sakai M, Koda T, Iwahori M, Kawano J. Internal porosity in denture base resin polymerized by microwave irradiation. J Gifu Dent Soc 1989;16:220-31.
21. Yunus N, Harrison A, Hugget R. Effect of microwave irradiation on the flexural strength and residual monomer levels of an acrylic resin repair material. J Oral Rehabil1994;21:641-8.

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