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ORIGINAL ARTICLE

Effect of drying different inclusion plasters on the mechanical properties of thermoactivated acrylic resins

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Abstract

This article aimed to evaluate some mechanical and chemical properties of acrylic thermoactivated resins by microwave energy, varying the condition and type of plaster. The groups were divided into Lucitone and Vipi-Wave groups, with or without previous treatment (drying) of type III and IV plasters. It was evaluated flexural strength, microhardness, roughness, porosity, residual monomer, and also, time and temperature relationship of plaster and acrylic resin during the polymerization cycles. The data were analyzed using Analysis of Variance (ANOVA) 5%, followed by Tukey's test. The results showed that the drying of the plaster influenced the results and the groups with dry plaster maintained a higher temperature permanence. Therefore, changes in the water condition contained in the inclusion plaster showed effects on the final properties of the acrylic resin, which may be a technical indicator for laboratory procedures in the manufacture of prosthetic devices.

Keywords: *acrylic resin, dental plaster, flexural strength, hardness, roughness.*

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1. Introduction

Polymethylmethacrylate (PMMA) or better known as acrylic resin is a polymer widely used in Dentistry. Although there are several clinical applications of acrylic resin, from temporary crowns to total prosthesis, PMMA has some limitations, such as low mechanical resistance[1] and dimensional change[2]. To improve these properties, several studies have been studying materials that can reinforce acrylic resin, as well as nylon, fiberglass, nanoparticles, among others[1,3], as well as ways to improve the processing technique of acrylic resin[4].

The mechanical properties can be influenced by the type of polymerization, for instance, by the water bath or by the autoclave[5, 6]. However, according to some studies, the best technique used is microwave polymerization, which produces greater mechanical resistance and less dimensional change when compared to the heat technique, but unlikely, a lower surface hardness[4,7]. The microwave is an alternative to the conventional method being easier to handle and clean because it warms up faster. Also, the activation is homogeneous, leading to a better finish of the prostheses[4,8].

The dimensional change occurs during the entire process, including as a result of choosing the type of plaster used during obtaining the work[2]. Another concern is the amount of residual monomer from the polymerization which irritates the patient's mucosa[9]. However, according to the work of Paes-Junior et al.[10] it was possible to reduce the amount of residues by pre-drying the plaster before curing the resin. During the separation of the plaster, depending on the agent used, a smoother and brighter surface is obtained[11]. Besides, to reduce the undesirable effects of PMMA, a light- and heat-cured urethane dimethacrylate (UDMA) emerged, which is characterized by its biocompatibility, low bacterial adherence, greater resistance to masticatory loads, and thus providing greater satisfaction for the patient[12].

Finally, the objective of the present study was to evaluate the mechanical properties of the acrylic resin by drying two types of plasters (III and IV) which were used in the inclusion phase, and also the activation method, by microwave oven or water bath. The null hypothesis was that the type and performance of dried plaster and the type of thermal activation did not influence the mechanical and chemical properties of the acrylic resin.

2. Materials and Methods

The materials used in the study were the following acrylic resins, Lucitone 550 (Dentsply Sirona, United States) and Vipi-Wave (Dental Vipi, Brazil) and also, the dental plasters, Gypsum stone type III Herodont (Coltene, Brazil)

and Gypsum stone type IV Velmix (Kavo Kerr, Brazil). The groups of the present study can be seen in Table 1.

Three types of metallic patterns of stainless steel with geometric shape of a parallelogram and dimensions respectively 2.0X2.0X2.0cm, 2.0X2.0X1.0cm, and 2.0X2.0X0, 5cm were used to perform the specimens. These patterns were copied by a laboratory silicone-based impression material (Rodorsil-VWL, Brazil), and from these replicas were made in wax 7 (Classic, Brazil).

The wax replicas were included in the muffle varying the type of the plaster and its manipulation. The type III plaster was mixed in the proportion of 180g of powder to 60ml of water. For the type IV plaster, the proportion was 180g of powder to 36 ml of water. Six wax patterns were positioned equidistantly and parallel to each other. The muffle was closed to leak the plaster into the counter muffle. The counter muffle was filled with plasters type III and IV provided in 240g of powder for 80ml of water and 240g of plaster for 50ml of water respectively, through the opening in the upper part of the muffle. For the groups where the plaster was previously dried, the procedure adopted was based on the methodology described by Canay et al.[13] and adapted by Paes-Junior et al.[10] who dried the inclusion plaster in a microwave oven for 10 min at 600W. Then, the muffles were left at room temperature for a period of one hour until completely cooled, and then with their parts open, they were stored for a minimum of 24 hours in a dry oven under temperature of 37ºC, before pressing and polymerizing acrylic resins. After one hour, the muffle parts were opened and the wax patterns were removed under immersion in heated water. Then, a thin layer of insulator for acrylic resin Al-Cote (Dentsply, Brazil) was applied with a brush over the entire surface area of the plaster. For the pressing procedure, Lucitone 550 or Vipi-Wave acrylic resin was used, being accommodated during the plastic phase in the spaces left by the wax.

Then, the muffle was closed and placed in a hydraulic press (Techno Máquinas, Brazil), until the final pressure of 1000Kgf maintained for 30 minutes before polymerization. For the microwave polymerization cycle was used a Continental AW-30 oven (BS Continental da Amazônia Ind. e Com. Ltda, Brazil) with a rotating plate and frequency of 2450 megahertz (MHz) with maximum power of 900W. The utilized cycle was 20% of the power of the device for 20 minutes plus 5min to 60% of the power. Then, the muffle was kept for about two hours at room temperature until cooled. To finish the pieces, a rotary sander (Panambra, Brazil) was used under constant refrigeration, and the wear was performed by the aluminum oxide sandpaper Norton (Norton), with decreasing weight (180, 320, 600#).

2.1. Fabrication of specimens for flexural strength and FTIR

Metallic patterns made in bars shape, rectangular, with sharp edges, in the dimensions of 27 x 12.60 x 3.10 mm were included in the muffle as described before. The only difference was the use of the Zetalabor silicone (Zermack, Brazil) which was applied around the metallic patterns. After 30 minutes, the counter muffle was settled and the plaster was poured. One hour after the final inclusion phase, the muffle was opened and the metallic patterns were removed. A thin layer of insulator was applied over the silicone and the acrylic resin was included in the muffle. After its polymerization, the specimens were removed from the muffle and placed in a water container, then the finishing was performed as already described. The FT-IR was performed in a PerkinElmer Spectrum One spectrophotometer, using the attenuated total reflectance (ATR) technique, in the 500-2000 cm^{-1} region, with a resolution of 4 cm^{-1} and 16 scans, in which are obtained the quantified MR performing the respective scans per specimen. The spectra obtained were sent to a software for graph analysis (Origin 7.0 with PeakFitting), where they were imported, analyzed, plotted and the comparative graphs established[14,15].

2.2. Microhardness analysis

Before the test, each group was stored in distilled water at a temperature of 37ºC+2ºC, for a period of 48h+2h. After this period, the microhardness analysis was performed by Vickers indentation test (VHN), using a microdurometer (FM 700, Japan), with a load of 10gf for 5 seconds, with 3 indentations in each specimen.

2.3. Porosity analysis

The results were obtained through the visual inspection by three previously calibrated observers who made a ranking and assigned scores based on the following criteria: 0 (without porosity), 1 (minimum amount of porosity), 2 (average amount of porosity), and 3 (large amount of porosity). After the evaluation, the arithmetic means of the scores attributed individually by the examiners were obtained.

2.4. Roughness analysis

The roughness analysis was performed over one of the faces of the specimen. However, before the analysis, the samples were ultrasonically cleaned with distilled water

Table 1. Groups analyzed according to the treatment of choice.

Groups	Treatment
LP	Lucitone 550 acrylic resin, microwave polymerization without prior treatment of type III plaster.
LPS	Lucitone 550 acrylic resin, microwave polymerization with pre-treatment of type III plaster (dissected plaster).
VP	Vipi-Wave acrylic resin, microwave polymerization without prior treatment of type III plaster.
VPS	Vipi-Wave acrylic resin, microwave polymerization with pre-treatment of type III plaster (dissected plaster).
L4	Lucitone 550 acrylic resin, microwave polymerization without previous treatment of type IV plaster.
L4S	Lucitone 550 acrylic resin, microwave polymerization with previous treatment of type IV plaster (dissected plaster).
V4	Vipi-Wave acrylic resin, microwave polymerization without prior treatment of type IV plaster.
V4S	Vipi-Wave acrylic resin, microwave polymerization with pre-treatment of type IV plaster (dissected plaster).

for 380s, and dried with an air jet, free of water and oil. For the roughness test, the specimens were analyzed in a contact roughness (Mitutoyo SJ – 400), with 3 readings of 4 mm, in two directions perpendicular to the sample surface. Roughness analysis was performed on one of the faces of the cubes used in the porosity analysis on one of the faces.

2.5. Flexural strength analysis

The samples were placed in distilled water and kept at a temperature of 37 ºC + 2ºC, for a period of 48h + 2h. After this period, the three-point flexural test was performed by a universal testing machine – EMIC (Model DL-1000, Brazil), with a load cell of 100Kgf and an application speed of 5 mm/min. The flexural strength values for the groups were obtained in mega Pascals (MPa) and subjected to statistical analysis.

2.6. Measurement of temperature in the polymerization cycle

A muffle from each group was subjected to the analysis of the temperature gradient. For this purpose, a device in the form of a metallic tablet was inserted into one of the thickest specimens (8 cm^3), surrounded by a plastic for its protection. According to the study by Savirmath and Mishra[16], dimensional change occurs with temperature change. These were positioned during the resin inclusion phase. The temperature monitoring was done 20 times, minute by minute of phase 1 of the polymerization cycle and, five times of phase 2 of the cycle.

An iButton DS1922E temperature sensor (Maxim integrated, USA) was used to perform the temperature measurement, which is a resistant and self-sufficient system that measures temperatures between 15ºC and 140ºC. This sensor is configured to communicate with a computer using the 1-Wire serial protocol, which connects to a USB port. The sensor is connected to this device which can program and activate according to the registration data, then the results are transferred to a computer in graphs and tables. This analysis was qualitative and determined a time/temperature graph for each group.

2.7. Remaining residual monomer (RM) analysis

The acrylic resin samples used in the flexural test were used. The specimens were submitted to finishing with 600 grit sandpaper and, five of each group had the MR quantified, through FT-Raman spectroscopy, with the respective scans per specimen. The obtained spectra were sent to a software (Origin 7.0 with Peak Fitting) which graphs were imported, analyzed, and established.

2.8 Statistical analysis

The obtained values were subjected to the normality and homogeneity test. For flexural strength, microhardness, and surface roughness, it was performed the ANOVA variance analysis with the Turkey test with a significance level of 5%. The porosity analysis did not follow a normal distribution so the non-parametric Kruskal-Wallis test was performed.

3. Results and Discussions

3.1. Porosity analysis

Regardless of the plaster, the type of resin, and drying (volume of 2 cm^3), the amount of porosity was practically non-existent for all groups. For the drying (volume of 4 cm^3), it was found slightly higher values for the LC and L4S groups. While for the drying (volume of 8 cm^3), the LC, LCS, and VCS groups had higher porosity than the other groups (Table 2). The groups that used Vipi-Wave resin had lower porosity values, being that the VP group showed absence of porosities regardless of the specimen volume. The VC and VPS groups showed minimal bubbles for the 8cm^3 volume and absence for the other volumes. This group was in the opposite direction to the others, where plaster drying, especially for specimens of greater volume, increased the occurrence of bubbles, which may perhaps be explained by the characteristics of the type III alpha hemidrate plaster used in this group, however there was no statistical difference between drying and the type of plaster.

3.2. Microhardness analysis

Despite dried processo, the groups that used type IV plaster showed lower values of microhardness compared to type III plaster. Regarding resin, Lucitone presented higher microhardness values (Table 3).

3.3. Roughness analysis

Type III plaster showed the lowest roughness, and when the plaster was previously dried, it also found small roughness values (Table 4), with the Lucitone resin showing lower values (Table 5).

3.4. Flexural strength analysis

Lucitone 550 resin presented greater flexural strength than Vipi- Wave resin and the drying of the plaster also influenced the increase of its resistance (Table 6).

Table 2. Kruskal-Wallis for the groups and volumes observed.

Volume	LC	LCS	LP	LPS	VC	VCS	VP	VPS	L4	L4S	V4	V4S
2mm	1	1	0	0	0	0	0	0	0	1	0	0
4mm	2	1	0	1	0	1	0	0	1	2	1	0
8mm	3	3	2	2	1	3	0	1	3	2	2	2

LC: Lucitone, without prior treatment of type II plaster. LCS: Lucitone, with pre-treatment of type II plaster. LP: Lucitone, without prior treatment of type III plaster. LPS: Lucitone, with pre-treatment of type III plaster. VC: Vipi-Wave, without prior treatment of type II plaster. VCS: Vipi-Wave, with pre-treatment of type II plaster. VP: Vipi-Wave, without prior treatment of type III plaster. VPS: Vipi-Wave, with pre-treatment of type III plaster. L4: Lucitone, without prior treatment of type IV plaster. L4S: Lucitone, with pre-treatment of type IV plaster. V4: Vipi-Wave, without prior treatment of type IV plaster. V4S: Vipi-Wave, with pre-treatment of type IV plaster.

Table 3. Vickers microhardness averages, considering only the type of plaster.

Plaster	Hardness		Resin	Hardness	
Type IV	24.82 ± 2.93	A	**Vipiwave**	25.85 ± 3.14	A
Type III	27.49 ± 4.11	B	**Lucitone**	27.74 ± 4.09	B

Table 4. Roughness averages (Ra), considering only the type of plaster.

Plaster	Roughness (Ra)		Drying	Roughness (Ra)	
Type III	0.14 ± 0.03	B	**Yes**	0.15 ± 0.03	A
Type IV	0.16 ± 0.03	A	**No**	0.16 ± 0.04	B

Table 5. Average roughness of the type of resin and whether or not drying was performed.

Drying	Resin		
Yes	Lucitone	0.14 ± 0.03	B
Yes	Vipiwave	0.16 ± 0.02	A
No	Vipiwave	0.16 ± 0.04	A
No	Lucitone	0.16 ± 0.04	A

Table 6. Flexural strength averages in MPa of the types of resin.

Resin	Strength		Drying	Strength	
Vipi-Wave	102.80 ± 15.33	A	**No**	107.55 ± 17.50	A
Lucitone	121.16 ± 31.06	B	**Yes**	116.41 ± 32.02	B

3.5. FTIR spectroscopy analysis

The FTIR curves of the Vipi- Wave and Lucitone resins can be interpreted in Figure 1 and 2, respectively, where it was observed that all the graphics obtained, regardless of the group, were coherent and were in the same observation ranges. In Figure 1, the curve corresponding to the band at 1150^{-1cm} was related to vibration and it was possible to infer that the VP group had a lower conversion when compared to the other groups. While in Figure 2 in the 750^{-1cm} band, the L4 and L4S groups had lower values.

3.6. Temperature gradient

Table 7 shows that for all groups there was a fast temperature rise of the resin when activated in the microwave, maintaining the temperature at the maximum peak, and decreasing quickly. When the plaster was dried, all the groups had a significant increase in the maintenance temperature, except for the LC group.

3.7. Discussion

The type of plaster, plaster drying method, and type of activation influenced the mechanical and chemical properties of the acrylic resin so the null hypothesis was rejected. Type III and IV plaster differ from each other in terms of the amount of water needed for their handling, so their mechanical strength was also different. In the present work, the plaster drying method was effective as it promoted a better removal of the water, without interfering with the subsequent inclusion and polymerization processes, in agreement with the works of Canay et al.[13]. This study evaluated the effect of the drying method of a type III plaster in a microwave and a conventional activation, which obtained an increase in resistance when the drying method was performed, however, there was no difference between the activation method of choice. In contrast, Paes-Junior et al.[10] analyzed whether the performance of drying plaster previously would influence the amount of residual monomer and the study found that the specimens where the

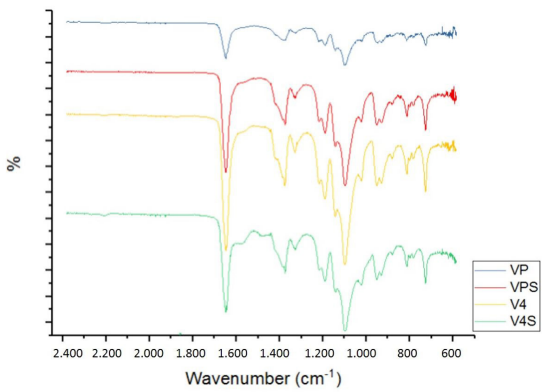

Figure 1. FTIR curve for the Vipi-Wave group.

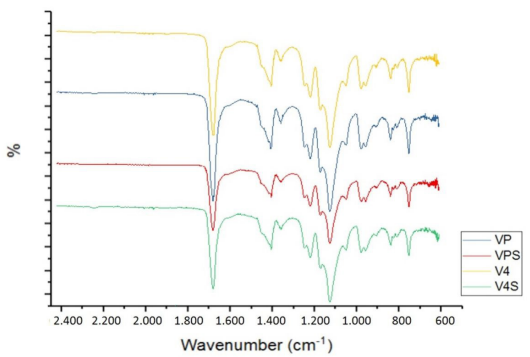

Figure 2. FTIR curve for the Lucitone group.

Table 7. Maximum temperature data (ºC) for the cycles and time of permanence for the experimental groups.

Temperature (ºC)	LC	LCS	LP	LPS	VC	VCS	VP	VPS
Temperatura peak	123°C	126°C	126°C	126°C	120°C	126°C	126°C	126°C
Permanence time	30s	30s	150s	300s	30s	300s	90s	360s

LC: Lucitone, without prior treatment of type II plaster. LCS: Lucitone, with pre-treatment of type II plaster. LP: Lucitone, without prior treatment of type III plaster. LPS: Lucitone, with pre-treatment of type III plaster. VC: Vipi-Wave, without prior treatment of type II plaster. VCS: Vipi-Wave, with pre-treatment of type II plaster. VP: Vipi-Wave, without prior treatment of type III plaster. VPS: Vipi-Wave, with pre-treatment of type III plaster.

drying method was performed obtained a lower amount of residual monomer.

The samples that had a thickness of 8 cm^3 showed a significant difference in porosity when compared to the volumes of 2 cm^3 and 4 cm^3, regardless of the plaster choice and drying. This probably occurred, because heat dissipation is harder on thick specimens thus causing internal porosities, also found in the work of Kimpara et al. [17] who evaluated four different polymerization cycles and found that performing short cycles has a greater amount of porosity. While Neisser et al.[18] also studied four different polymerization cycles and observed that the shorter cycles with lower energy, raised the temperature, thus causing a higher index of porosity.

The Ibutton wireless device showed a fast increase of temperature, with a constant temperature peak of the specimens for all groups, especially in the groups where plaster was dried due to the slow heat dissipation. Regarding the FTIR test to evaluate the degree of polymerization, a qualitative aspect was considered where all the obtained graphs, regardless of the group, had the same observation ranges.

In the analysis of roughness, the LPS group showed a higher level of roughness, regardless of the volume of the material used. In the study performed by Rizzatti-Barbosa and Ribeiro-Dasilva[19], it was compared the roughness of the simultaneous polymerization or double vial of the upper and lower total dentures that were made using the microwave or conventional technique. It was observed that there were no differences between the groups studied.

For the polymerization of acrylic resin, it is necessary to activate free radicals, this can occur through microwave waves, heat by hot water, or even by photoactivation. In the literature, the use of the microwave technique for polymerization reduced the amount of residual monomer present in total dentures[20]. In the present study, a significant change was observed in the flexural strength for the drying method performed in the plaster, when compared to the conventional cycle in a heated water bath, in agreement with the research by Silva et al.[7]. This study evaluated the flexural strength of four heat cycles by microwave energy and the authors concluded that when the microwave was used, the flexural strength was greater when compared to the other groups.

4. Conclusions

Within the limitations of this study, it was possible to conclude that the drying process of the plaster, regardless of the type of plaster used, could influence the mechanical properties of the thermoactivated resins presented in this study.

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6. References

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