Study of the surface hardness and modulus of elasticity of conventional and microwave-cured acrylic resins

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Abstract: The aim of this study was to evaluate the following acrylic resins: Clássico®, QC-20® and Lucitone®, recommended specifically for thermal polymerization, and Acron MC® and VIPI-WAVE®, made for polymerization by microwave energy. The resins were evaluated regarding their surface nanohardness and modulus of elasticity, while varying the polymerization time recommended by the manufacturer. They were also compared as to the presence of water absorbed by the samples. The technique used was nanoindentation, using the Nano Indenter XP®, MTS. According to an intra-group analysis, when using the polymerization time recommended by the manufacturer, a variation of 0.14 to 0.23 GPa for nanohardness and 2.61 to 3.73 GPa for modulus of elasticity was observed for the thermally polymerized resins. The variation for the resins made for polymerization by microwave energy was 0.15 to 0.22 GPa for nanohardness and 2.94 to 3.73 GPa for modulus of elasticity. The conclusion was that the Classico® resin presented higher nanohardness and higher modulus of elasticity values when compared to those of the same group, while Acron MC® presented the highest values for the same characteristics when compared to those of the same group. The water absorption evaluation showed that all the thermal polymerization resins, except for Lucitone®, presented significant nanohardness differences when submitted to dehydration or rehydration, while only Acron MC® presented no significant differences when submitted to a double polymerization time. Regarding the modulus of elasticity, it was observed that all the tested materials and products, except for Lucitone®, showed a significant increase in modulus of elasticity when submitted to a lack of hydration.

Descriptors: Denture, complete; Polymethyl methacrylate; Hardness; Elasticity.

Introduction

For many decades, polymethylmethacrylate has been the first choice for making the base of complete dentures. Thermally activated acrylic resins have been traditionally processed by immersion in temperature-controlled water, for a specific period of time.[1] Recently, polymerization methods involving activated light and microwave energy were introduced.

In the last years, many studies have been carried out in order to improve the physical and mechanical properties of the acrylic resins activated by microwave energy.[1-6] The heating by microwave energy is independent on thermal conductivity. When dielectric materials are placed in a microwave energy field, some kind of molecular friction takes place and these materials warm up rapidly.[3] Some authors believe that physical proprieties such as hardness and transversal resistance are favored when this process polymerizes the resins,[4] others believe that the polymerization by immersion in heated water during a long cycle results in better physical properties.[2] Others still consider that the conventional and microwave energy methods are similar, such as Smith *et al.*[5] (1992), who submitted some materials to the existing polymerization methods: conventional (water heat diffusion), microwave energy and light, and then compared properties such as hardness, surface smoothness, transversal resistance, modulus of elasticity and resistance to impact. The only difference found between the two methods was that the second showed higher values for modulus of elasticity.

When the physical properties of the acrylic resins are highlighted, many are the elements that can vary inside its structure, either cooping or creating obstacles that are liable to interfere with the material's ultimate rate of success. Among these elements we can point out the modulus of elasticity, which is directly related to the material's resistance to flexion, nanohardness, and also the loss modulus and fluidity, which are related to the material's viscosity and therefore with the probability of it suffering fractures or permanent deformations.[1]

In the present study the surface nanohardness and the modulus of elasticity *versus* variations on the polymerization time recommended by the manufacturer of five acrylic resins broadly used for the base of complete dentures were studied. The same characteristics were evaluated when the materials were submitted to a lack of hydration followed by rehydration.

Material and Methods

Manufacture of the samples made of resin polymerized by a thermal procedure

To manufacture the samples, glass slides measuring 10 mm x 10 mm x 2 mm were used. 100 g of Herodent® type IV dental stone (Vigodent, Rio de Janeiro, RJ, Brazil) were mixed to 24 ml of distillated water, which was then poured into a copper muffle used for conventional polymerization of resins, previously isolated with vaseline. The glass slides were then embedded in the muffle, and after the time recommended by the stone manufacturer for its complete crystallization, the same muffle was adapted to the counter muffle, which was completely filled with a new portion of stone. After its complete crystallization, the muffle was opened and the glass slides were removed.

The monomer and the polymer of the Clássico® resin (Clássico Artigos Odontológicos Ltda., São Paulo, SP, Brazil) were then mixed according to the proportion recommended by the manufacturer. When the mix reached the working phase, the material was embedded in the muffle. This way, faithful

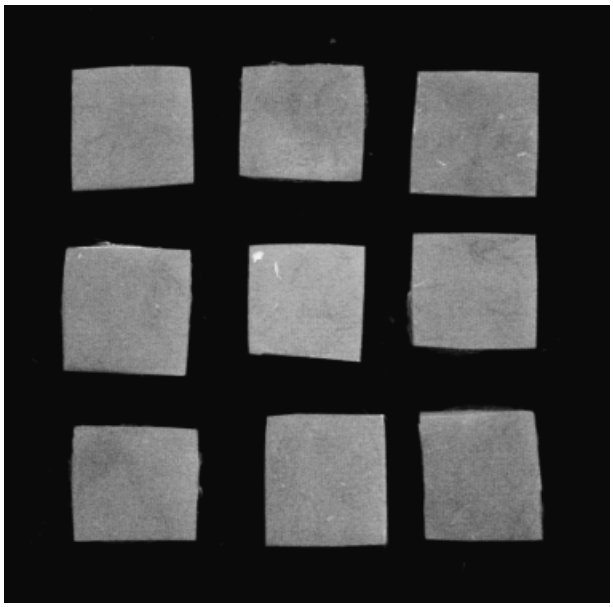


Figure 1 - Resin samples made for the readings.

copies of the samples were produced in resin (Figure 1). The force applied to close the muffle was 1,250 kgf, maintained by a hydraulic press.

The whole set rested for 4 hours and after that the polymerization cycle started. This was made inside an electrical polymerizing machine, with temperature regulated at 74°C, for 9 hours. After the resin was polymerized and the muffle cooled off, the sample bars were removed and polished with alumina, in order to obtain flat and smooth surfaces.

Identical procedures were performed in order to obtain samples of the resins Lucitone® (Dentsply Indústria e Comércio Ltda., Petrópolis, RJ, Brazil) and QC-20® (Dentsply Indústria e Comércio Ltda., Petrópolis, RJ, Brazil).

Manufacture of the resin samples polymerized by microwave energy

Once more a muffle was used in order to polymerize the resin samples. These muffles were now made of reinforced glass fiber (Figure 2), especially made for use in microwave ovens. Following the same technique as that used to obtain the samples of the previous resins, glass slides were then included in the muffles. The same Herodent® type IV dental stone was used.

The first resin to be manipulated was Acron MC® (GC Lab Technologies Inc., Alsip, IL, USA), following the manufacturer's instructions, and when the mix reached the working phase it was embedded in the muffle, which was closed with the aid of screws. The force used for closing was 1,250 kgf.

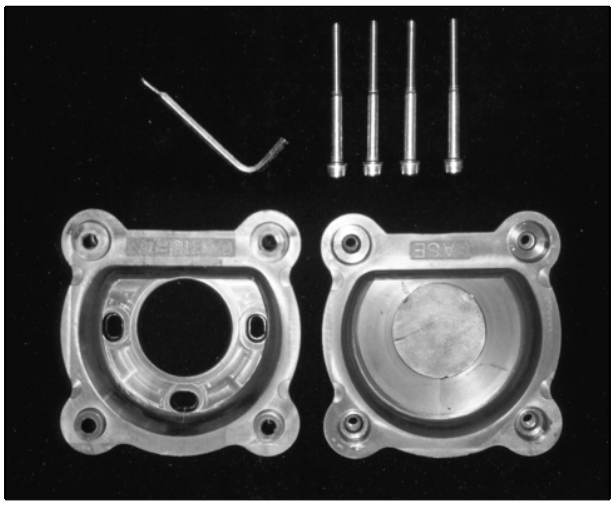


Figure 2 - Fiberglass muffle (GC brand, Dental Industrial Corporation) proper for microwave use.

After waiting 15 minutes the whole set was placed inside a Sharp® microwave oven, on the center of the plate, and was irradiated for 3 minutes, with 500 W of power. A 20 minutes break was taken and the muffle was washed with cool water on the outside. After 20 more minutes its complete cooling was checked and only then the samples were removed from its interior. The same procedure for polishing described for the previous group was carried out.

Three different groups of samples were obtained. One of them was polymerized following exactly the time and procedures recommended by the manufacturer. The other two were polymerized with twice and half of the polymerization time recommended, respectively, for the Acron MC®resin.

These procedures were repeated for the VIPI-WAVE® resin (VIPI Ltda., Pirassununga, SP, Brazil) according to the manufacturer's recommendations, which state that the mixture should be taken to a microwave oven with 900 W of power, and be irradiated with 40% of the power for 2 minutes, and then rest for 15 minutes. After the resting period, they should be irradiated again with 20% of the power for 8 minutes, and finally for 3 minutes with 60% of the power. This way, a standard group was obtained, polymerized by following precisely the manufacturer's directions. Another two groups were irradiated, respectively, with twice as much time and half of the time recommended.

All the samples were always kept immersed in distillated water.

Resin samples analysis and reading

To read the samples a nanoindentation device named Nano Indenter XP (MTS Nano Instruments, Oak-Ridge, TN, USA) was used. This device is made of three different parts: a penetrating cylinder head, an optical microscope and a mobile table (Figure 3).

The samples were placed inside the device in order to be read, following a time schedule according to the type of resin obtained and the polymerization time used for each polymerization (Figure 4).

Before all the analyses were done, the behavior

Figure 3 - Assessment device of the Nano Indenter XP.

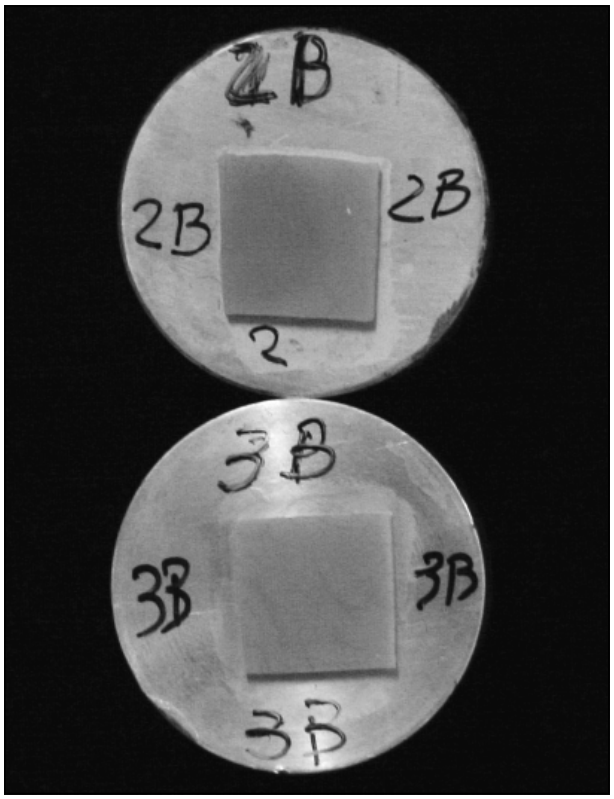


Figure 4 - Samples made for the measurements.

of the resins was observed after the first reading, and after 7 days of dehydration, after which a complete set of readings was performed. This was called the 2nd reading. The samples were then rehydrated for another 7 days, and then the 3rd reading was performed inside the Nano Indenter XP®.

All the readings in this study were accomplished by 8 loads of indentations, each one of them having a set of approximately 13 readings, completing an average of one hundred reading tests for each sample total, going from zero to 7 thousand nanometers of depth.

Results

As we analyzed the nanohardness of the conventional, thermally polymerized resins, which were polymerized according to the recommendations of the manufacturer, it was observed that the Clássico resin presented a significant difference when compared to QC-20® and Lucitone®, which were very similar to each other (Table 1).

The same occurred to these materials when considering their modulus of elasticity (Table 2).

The group of resins that were polymerized by microwave energy according to the standard time of polymerization presented significant differences. Acron MC® presented higher nanohardness and modulus of elasticity values than VIPI-WAVE® (Table 1).

When comparing the two groups submitted to different polymerization methods, it was observed that there was a similarity of results, both regarding nanohardness and modulus of elasticity, especially when comparing Acron MC® and Clássico, and VIPI-WAVE® and QC-20®/Lucitone®.

Regarding the values of modulus of elasticity *versus* variations in the polymerization time, it was observed that, for Acron MC®, as this time decreased, the modulus of elasticity also decreased, and when the time was doubled, the modulus of elasticity also showed an increase, although keeping a level which was similar to that obtained with the standard polymerization time.

The VIPI-WAVE® resin presented an increase in modulus of elasticity when compared to that obtained when the standard polymerization was performed (according to the manufacturer's recommendations), both with half of the polymerization time and with the polymerization time doubled.

Regarding the resins' behavior when submitted to a lack of hydration (2nd reading), and to an ensuing rehydration (3rd reading), it was observed that in the conventional thermal polymerization group, the values for nanohardness were very similar when us-

Table 1 - Descriptive statistics for type of material / Hardness (GPa).

Data			Average	Standard Deviation	Minimum	Maximum	P value[1]	
Clássico®		Standard	0.236	0.016	0.222	0.264	< 0.0001	S
		Without water	0.234	0.005	0.226	0.240		
		With water	0.207	0.007	0.196	0.216		
Lucitone®		Standard	0.156	0.008	0.147	0.169	0.084	NS
		Without water	0.162	0.009	0.152	0.177		
		With water	0.158	0.007	0.145	0.168		
QC-20®		Standard	0.145	0.017	0.123	0.170	0.008	S
		Without water	0.155	0.017	0.136	0.178		
		With water	0.147	0.015	0.136	0.177		
VIPI - WAVE®	Standard time	Standard	0.158	0.009	0.149	0.175	< 0.0001	S
		Without water	0.193	0.015	0.175	0.216		
		With water	0.175	0.018	0.157	0.204		
	Half the Standard time	Standard	0.255	0.013	0.241	0.278	< 0.0001	S
		Without water	0.295	0.035	0.259	0.350		
		With water	0.291	0.028	0.259	0.338		
	Twice the standard time	Standard	0.243	0.007	0.235	0.254	< 0.0001	S
		Without water	0.282	0.029	0.257	0.330		
		With water	0.268	0.019	0.247	0.298		
Acron MC®	Standard time	Standard	0.226	0.007	0.219	0.238	< 0.0001	S
		Without water	0.326	0.034	0.285	0.365		
		With water	0.231	0.031	0.192	0.276		
	Half the Standard time	Standard	0.191	0.024	0.167	0.238	< 0.0001	S
		Without water	0.198	0.020	0.177	0.230		
		With water	0.169	0.015	0.149	0.190		
	Twice the standard time	Standard	0.284	0.034	0.247	0.336	0.328	NS
		Without water	0.282	0.010	0.265	0.298		
		With water	0.273	0.014	0.250	0.286		

(1) Intra-group analysis, using a parametric test (Repeated Measures ANOVA).

ing the standard time of polymerization, except for a few variations, as can be seen in Graph 1.

Yet in the group of resins polymerized by microwave energy there was a variation in nanohardness values when they were submitted to dehydration, specially for Acron MC®, when they were polymerized according to the time recommended by the manufacturer. This variation, however, diminished after rehydration. The same happened to their modulus of elasticity (Graph 2).

Discussion

The resins used for the base of complete dentures are of utmost importance, since the maximum bite strength of a patient using a complete denture is approximately 1/6 of that of a dentate patient. For this reason, it is very important that its adaptability, structure and especially manipulation procedures be reliable.

Currently, the focus of researchers regarding microwave energy has been the analysis of the polymerization method. Nevertheless, there is some con-

Table 2 - Descriptive statistics for type of material / Modulus of Elasticity (GPa).

Data			Average	Standard Deviation	Minimum	Maximum	P value[1]	
Clássico®		Standard	3.724	0.147	3.591	3.962	< 0.0001	S
		Without water	3.709	0.084	3.628	3.841		
		With water	3.466	0.094	3.350	3.646		
Lucitone®		Standard	2.925	0.200	2.757	3.386	0.872	NS
		Without water	2.924	0.061	2.862	3.044		
		With water	2.894	0.072	2.785	2.983		
QC-20®		Standard	2.613	0.168	2.415	2.857	0.008	S
		Without water	2.716	0.165	2.539	2.941		
		With water	2.733	0.190	2.594	3.161		
VIPI - WAVE®	Standard time	Standard	2.945	0.080	2.870	3.072	< 0.0001	S
		Without water	3.282	0.095	3.202	3.467		
		With water	3.148	0.154	2.990	3.412		
	Half the Standard time	Standard	3.864	0.108	3.746	3.994	< 0.0001	S
		Without water	4.128	0.210	3.883	4.434		
		With water	4.217	0.230	3.991	4.546		
	Twice the standard time	Standard	3.808	0.080	3.741	3.945	< 0.0001	S
		Without water	4.076	0.151	3.923	4.367		
		With water	4.079	0.150	3.905	4.299		
Acron MC®	Standard time	Standard	3.737	0.074	3.619	3.815	< 0.0001	S
		Without water	4.634	0.107	4.506	4.786		
		With water	3.688	0.187	3.424	3.949		
	Half the Standard time	Standard	3.135	0.213	2.859	3.502	< 0.0001	S
		Without water	3.246	0.095	3.129	3.415		
		With water	3.030	0.142	2.870	3.254		
	Twice the standard time	Standard	4.048	0.190	3.838	4.370	0.927	NS
		Without water	4.069	0.081	3.958	4.214		
		With water	4.070	0.190	3.841	4.368		

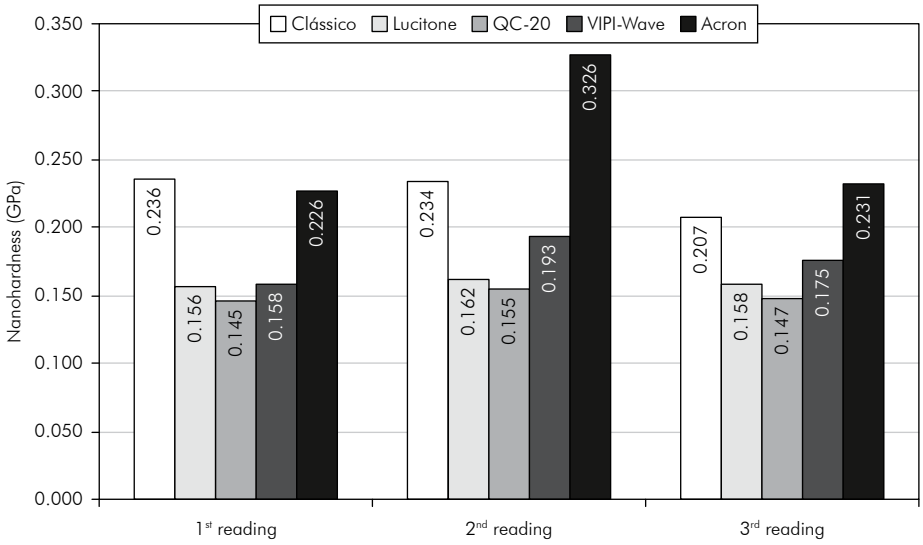
(1) Intra-group analysis, using a parametric test (Repeated Measures ANOVA).

cern about the types of resin being used, as in the study by Kimura *et al.*[3] (1983), who state that the resin should be carefully chosen in order to avoid fractures, or by Hayden[7] (1986), who also links the type of resin to its influence on the resistance to fractures. Both, however, end up comparing the polymerization methods, using microwave energy or a conventional thermal water bath.

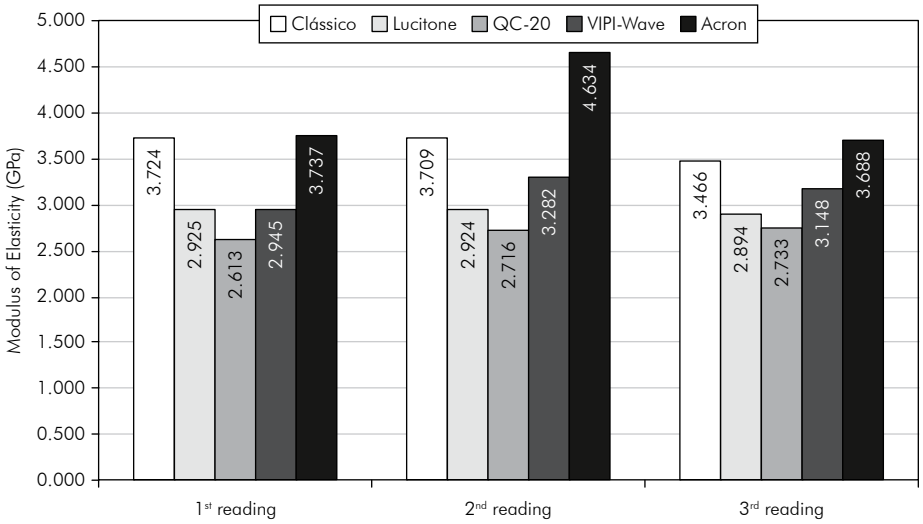
When varying the polymerization time, specifically when considering microwave resins, it was observed that, except for a very small variation with the Acron MC®resin when using half of the polymerization time, there was no significant difference for all the attempts, and no significant difference was observed when this time was changed, according to the depths of the samples.

These data are in accordance with the studies by Sanders *et al.*[8] (1987), De Clerck[9] (1987), Levin *et al.*[10] (1989), Shlosberg *et al.*[11] (1989), Ilbay *et al.*[12] (1994), Arima *et al.*[13] (1995), Monteiro Netto *et al.*[14] (1999) and their concerns regarding the resistance to fracture of the materials when submitted to

Graph 1 - Nanohardness of the resins in all groups.



Graph 2 - Modulus of elasticity of all resins in all groups.



masticatory loads. In order to achieve good results, an extra effort should be made to guarantee proper manipulation of the materials.

Conclusion

The results obtained under the described conditions allow us to conclude that the Clássico® resin presented higher nanohardness values and a higher modulus of elasticity when compared to its congeners, while Acron MC® presented higher values of nanohardness and modulus of elasticity in the microwave energy polymerization group. When varying the polymerization time, only Acron MC® presented no difference when submitted to twice the polymerization time.

Regarding the behavior of the resins when submitted to a lack of humidity (2nd reading) and subsequent rehydration (3rd reading), the resins polymerized by microwave energy presented some hardness variations when dehydrated, specially Acron MC®, when following the standard time of polymerization recommended by the manufacturer. These variations, however, diminished after rehydration.

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