Effect of Pressure, Post-Pressing Time, and Polymerization Cycle on the Degree of Conversion of Thermoactivated Acrylic Resin

Rafaela de S. Leão, Sandra L. D. de Moraes, Kátia A. da S. Aquino, Cristina P. Isolan, Bruno G. da S. Casado, and Marcos A. J. R. Montes

1Department of Restorative Dentistry, Faculty of Dentistry, University of Pernambuco-FOP/UPE, Av. Gal. Newton Cavalcanti 1650, 54753-020 Camaragibe, PE, Brazil
2Department of Prosthodontics, Faculty of Dentistry, University of Pernambuco-FOP/UPE, Av. Gal. Newton Cavalcanti 1650, 54753-020 Camaragibe, PE, Brazil
3Department of Nuclear Engineering, Federal University of Pernambuco (UFPE), Av. Prof. Moraes Rego, 1235-Cidade Universitária, 50670-901 Recife, PE, Brazil
4Department of Restorative Dentistry, Faculty of Dentistry, Federal University of Pelotas (UFPe), R. Gonçalves Chaves, 457-Centro, 96015-560 Pelotas, RS, Brazil

Correspondence should be addressed to Rafaela de S. Leão; rafaellaleao90@hotmail.com

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Herein, the effect of different post-pressing times and pressure in two cycles of polymerization on the degree of conversion (DC) of thermally activated acrylic resin (TRRA) is analyzed to optimize the polymerization of this material. After post-pressing for 0, 6, or 12 h, polymerization was performed with or without a pressure of 60 psi (0.41 MPa) in a short (4 h) or a long (11 h) cycle, totaling 12 groups. To determine the DC, PMMA specimens were analyzed by Fourier transform infrared spectroscopy. The influence of each factor alone on the DC was studied by experimental planning. The statistical tests used were three-way ANOVA, t-test, Tukey’s test, and Levene’s test, with a margin of error of 5%. Two groups prepared with post-pressing times of 12 h had the lowest DC (p < 0.001). Post-pressing times of 0 and 6 h did not yield statistically different results. Pressure increased the DC in only one group (long cycle +12 h, p = 0.001). The short cycle resulted in a higher DC than the long cycle in 2 groups (with pressure +0 h, p = 0.002; without pressure +6 h, p = 0.015), while the long cycle yielded a statistically higher DC in only one group (with pressure +12 h, p < 0.001). The polymerization showed satisfactory DC in all 12 groups. Small differences found among the specimens indicate that the pressure, post-pressing time, and polymerization cycles herein were not influential factors for the DC of PMMA.

1. Introduction

Thermoactivated acrylic resins, such as poly (methyl methacrylate) (PMMA), are alloplastic materials used in dentistry and present ease of handling, mechanical resistance, good availability, low thermal and electrical conductivity, low weight, and low cost [1]. PMMA has been mainly used in removable total or removable partial bases since 1930 [2–5]. In addition, they are promising for fabricating internal prostheses and replacing missing bony segments of the skull and face [6–9]. Studies have found that PMMA yields results similar to gold standard materials such as autogenous bone and titanium [1] for these purposes, which makes this material one of the most versatile for the health industry.

Despite its advantages, adverse reactions to PMMA have been reported in the literature [10–12]. The viability of cells exposed to the acrylic resin is influenced mainly by the
presence of residual monomers in the samples, that is, by the
degree of conversion (DC) achieved in the polymerization
reaction [9, 13, 14].

In vinyl polymers, the DC is calculated from the ratio of
the concentration of aliphatic C=C double bonds remaining
in a polymerized sample to the total number of C=C bonds
in the unpolymerized sample, that is, the monomer of origin.
Fourier transform infrared spectroscopy (FTIR) is one of the
most widely used methods to evaluate the DC [15].

When processing dental prostheses, the most studied
parameter affecting DC is the polymerization cycle. Com-
mon polymerization methods are microwave energy, au-
toclave polymerization, and (the most used) water bath
[16, 17]. Polymerization can be realized using two cycles:
a short cycle and a long cycle [5]. A good acrylic poly-
merization method is capable of achieving the best physi-
cochemical and biological properties of the acrylic resin,
such as hardness, porosity, and monomer release [4].

The influence of other factors such as post-pressing time
and the use of pressure during polymerization have not yet
been established. Studies on the mechanical properties re-
ported improvements after 12 and 24 h of post-pressing
time, supposedly due to an increase of the DC [2, 16]. High pressures (500 MPa) positively influenced the trans-
formation of the monomers into high-molecular-weight
polymers by increasing the conversion percentage [18].
However, literary records proving the influence of smaller
pressures, which can be applied by typical laboratory-scale
equipment, are not yet available.

In view of the lack of information on the correlation of
post-pressing time and influence of the pressure on the DC,
this study aimed at analyzing the effect of these factors on the
conversion of PMMA in two cycles of polymerization. The
null hypotheses of this study are as follows: there is no
association between post-pressing time and polymerization
rate, and there is no association between presence of
pressure and degree of PMMA conversion.

2. Materials and Methods

2.1. Preparation of Specimens. The specimens were prepared
in the same way as in the manufacture of conventional total
prostheses. For this, aluminum magazines with the di-

mensions of 40 × 10 × 2 mm³ were manufactured. The base
of plastic flasks was partially filled with 150 g of type III
gypsum (Herodent, Vigodont Coltene). After gypsum
crystallization, the space left between the gypsum and the
edge of the base of the muffle was filled with laboratory-
grade silicone (Zetalabor, Zhermack), in which the matrices
were inserted.

The muffle was closed and a new layer of plaster was
poured over it. The resulting mold was filled with a Lucitone
199 acrylic resin at the powder/liquid ratio recommended by
the manufacturer (light pink powder: PMMA polymer,
pigments, and fiber aesthetics; liquid: methyl methacrylate;
Dentsply Industria e Comércio LTDA, Petrópolis, RJ,
Brazil). The muffle was closed and then pressed using a
hydraulic press (Reco Hydromatic Press-Reco Dental,
Wiesbaden); the applied pressure was slowly increased to
100 kgf·cm⁻² (9.8 MPa). Subsequently, the specimens were
either immediately polymerized (post-pressing time of 0 h)
or subjected to post-pressing for 6 h or 12 h prior to
polymerization.

The test specimens were polymerized in a pneumatic
digital polymerizer (PPD Indústria Digital 08 Fenix; Souza
and Marques Pedranópolis, SP, Brazil). The test groups were
polymerized with a short or long cycle. For the short cycle,
first, water was heated to 70°C (47 min) and this temperature
was maintained for 1 h 50 min. Subsequently, the water
temperature was raised to 100°C (5 min) and held for 1 h
5 min, leading to a total polymerization time of approxi-
mately 4 h. For the long cycle, water was heated to 70°C (40 ±
5 min) and then held at this temperature for 9 h. The water
temperature was then raised to 100°C (30 min) and held for
30 min, leading to a total polymerization time of approxi-
mately 11 h. To analyze the influence of applied pressure
before the polymerization, 60 psi (0.41 MPa) of pressure was
applied. Upon completion of the polymerization cycle, the
specimens were allowed to cool on the bench; the muffle was
then opened and the specimens were removed.

Thus, through the combinations of the studied variables,
12 types of samples were prepared (Table 1). The sample
calculation was performed using the program BioEstat 5.0,
based on the results of the mean and standard deviation of
two groups of 6 specimens each, as detailed in an article by
Bural et al. [13].

2.2. Analysis of the Degree of Conversion. The degree of
conversion of methyl methacrylate (MMA) to PMMA was
determined using Fourier transform infrared spectroscopy
in the attenuated total reflectance (FTIR-ATR) mode. A
Shimadzu Prestige 21 spectrometer (4 cm⁻¹ resolution and
16 scans) was used in the absorbance mode. All specimens as
well as the nonpolymerized material were analyzed.

To prepare the PMMA samples for the FTIR-ATR
analysis, the surface of each polymerized test specimen
was sprayed with a tungsten drill; the resulting powder was
sieved to obtain a uniform particle size and was placed on the
ATR crystal for the test. To prepare the nonpolymerized
material, the prepolymerized resin powder was heated at
70°C for 30 h to degrade the benzoyl peroxide and to avoid
polymerization reactions of the material after handling with
the (liquid) monomer. After mixing the powder (without
benzoyl peroxide) and liquid (monomer) following the
manufacturer’s instructions, the resulting blend was also
placed on the ATR crystal.

The DC was determined from the ratio of the absorbance
intensity observed for aliphatic C=C at 1635 cm⁻¹ and the
absorbance intensity of the carbonyl bond C=O (1720 cm⁻¹),
used as the internal standard of polymerized material for
both the polymerized material and the unpolymerized
material, according to the following equation [19]:

\[
\text{degree of conversion (\%)} = 100 \left[ 1 - \frac{(A_{1638}/A_{1720})_{\text{polymerized}}}{(A_{1638}/A_{1720})_{\text{unpolymerized}}} \right].
\]
2.3. Statistical Analysis. An experimental design was established to analyze the influence of each factor on the DC. Data were expressed as mean, standard deviation, and coefficient of variation and were inferentially analyzed by three-way ANOVA, one-way ANOVA F test, and Student’s t-test with equal or unequal variances. In the case of significantly different post-pressing times, Tukey’s multiple comparison test was used.

Equality of variances was assessed using the Levene test. The margin of error used in the statistical test decisions was 5%. The data were entered into an EXCEL worksheet, and the statistical calculations were performed with the statistical program SPSS (Statistical Package for the Social Sciences) version 23.

3. Results

Figure 1 shows a simple representation of the polymerization of MMA to PMMA. Figure 2 shows the obtained FTIR-ATR spectra of MMA and PMMA in the 1800–1500 cm\(^{-1}\) region. In Table 2, the main FTIR bands attributed to PMMA and MMA according to Silverstein et al. [20] are given.

The DC values (%) and \(p\) values obtained from the statistical analysis of the DC for each studied variable are presented separately in Tables 3–5. The lowest mean DC (92.66%) was recorded for the long polymerization cycle without applied pressure and with a post-pressing time of 12 h. The mean DC was higher (97.39%) when the post-pressing time was 0 h under otherwise identical conditions.

An ANOVA evaluation of the DC for the 12 groups studied with a factor considering the 12 combinations of the variables is presented in Table 6. By means of Tukey’s multiple comparisons test, we verified in which groups the DC mean difference occurred.

4. Discussion

This work presents some results that have not yet been reported in the literature. The null hypotheses of this study were accepted: neither the post-pressing time nor the presence or absence of pressure during the polymerization had a statistically significant influence on the DC of MMA to PMMA.

Despite the fact that few studies focus on the influence of pressure on PMMA, the results of this study can be correlated with those found in works by Murakami et al. [18] and Nguyen et al. [21]. It should be noted however that these studies did not use the same pressure conditions. Here, the
application of a pressure of 500 MPa resulted in the formation of high-molecular-weight polymers with a high DC. According to Nguyen et al. [21], this result was due to the decrease of intermolecular distance in the material, reducing the free volume and consequently generating a product with a greater density and a reduced number and size of defects. In the present work, we used a smaller pressure of 0.41 MPa due to limitations of our laboratory equipment, and we observed no significant influence of applying this pressure on the DC. Thus, a pressure of 0.41 MPa was not able to increase the molecular weight of PMMA and influence the DC; therefore, it was not possible to verify the statistical difference for the DC values of the majority of the groups, with and without pressure.

Our findings are in agreement with those of Lee et al. [22] who used pressures similar to ours to analyze chemically activated acrylic resins. Note that the same author pointed out that these pressures, while not affecting hardness and DC, could affect porosity. In view of the applications of this material, especially in internal prostheses where the residual

| Cycle | Pressure | 0 h Mean ± SD | 6 h Mean ± SD | 12 h Mean ± SD | p value |
|-------|----------|---------------|---------------|---------------|---------|
| Short | Without  | 96.30 ± 1.61 (1.67) | 97.34 ± 1.19 (1.22) | 93.09 ± 4.36 (4.68) | p = 0.045* |
| Short | With     | 96.55 ± 0.49 (0.51) | 95.95 ± 0.98 (1.02) | 93.74 ± 1.04 (1.11) | p < 0.001* |
| Long  | Without  | 97.39 ± 0.96 (0.99) | 95.43 ± 1.07 (1.12) | 92.66 ± 1.83 (1.97) | p < 0.001* |
| Long  | With     | 94.68 ± 1.01 (1.07) | 95.93 ± 2.20 (2.29) | 96.16 ± 0.58 (0.60) | p = 0.160 |

*Significant difference; p = probability of significance.

| Cycle | Pressure | 0 h Mean ± SD | 6 h Mean ± SD | 12 h Mean ± SD | p value |
|-------|----------|---------------|---------------|---------------|---------|
| Short | Without  | 96.30 ± 1.61 (1.67) | 97.34 ± 1.19 (1.22) | 93.09 ± 4.36 (4.68) | 0.045* |
| Short | With     | 96.55 ± 0.49 (0.51) | 95.95 ± 0.98 (1.02) | 93.74 ± 1.04 (1.11) | 0.001* |
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| Long  | With     | 94.68 ± 1.01 (1.07) | 95.93 ± 2.20 (2.29) | 96.16 ± 0.58 (0.60) | 0.001* |

*Significant difference; p = probability of significance.

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| Long  | With     | 94.68 ± 1.01 (1.07) | 95.93 ± 2.20 (2.29) | 96.16 ± 0.58 (0.60) | 0.001* |

*Significant difference; p = probability of significance.

| Cycle | Pressure | 0 h Mean ± DP (CV%) | 6 h Mean ± DP (CV%) | 12 h Mean ± DP (CV%) | p value |
|-------|----------|----------------------|---------------------|----------------------|---------|
| Short | Without  | 96.30 ± 1.61 (1.67) (AB) | 97.34 ± 1.19 (1.22) (AC) | 93.09 ± 4.36 (4.68) (AB) | 0.045* |
| Short | With     | 96.55 ± 0.49 (0.51) (ABC) | 95.95 ± 0.98 (1.02) (AB) | 93.74 ± 1.04 (1.11) (BE) | 0.001* |
| Long  | Without  | 97.39 ± 0.96 (0.99) (AC) | 95.43 ± 1.07 (1.12) (AB) | 92.66 ± 1.83 (1.97) (BD) | 0.001* |
| Long  | With     | 94.68 ± 1.01 (1.07) (AE) | 95.93 ± 2.20 (2.29) (AB) | 96.16 ± 0.58 (0.60) (AB) | 0.001* |

*Significant difference; p = probability of significance.

Letters represent statistical similarity (obs: consider only one letter, not necessarily the set of letters).

| Cycle | Pressure | 0 h Mean ± DP (CV%) | 6 h Mean ± DP (CV%) | 12 h Mean ± DP (CV%) | p value |
|-------|----------|----------------------|---------------------|----------------------|---------|
| Short | Without  | 96.30 ± 1.61 (1.67) (AB) | 97.34 ± 1.19 (1.22) (AC) | 93.09 ± 4.36 (4.68) (AB) | 0.045* |
| Short | With     | 96.55 ± 0.49 (0.51) (ABC) | 95.95 ± 0.98 (1.02) (AB) | 93.74 ± 1.04 (1.11) (BE) | 0.001* |
| Long  | Without  | 97.39 ± 0.96 (0.99) (AC) | 95.43 ± 1.07 (1.12) (AB) | 92.66 ± 1.83 (1.97) (BD) | 0.001* |
| Long  | With     | 94.68 ± 1.01 (1.07) (AE) | 95.93 ± 2.20 (2.29) (AB) | 96.16 ± 0.58 (0.60) (AB) | 0.001* |

Letters represent statistical similarity (obs: consider only one letter, not necessarily the set of letters).
monomer levels should be the lowest possible and good mechanical properties are essential, further studies are necessary to determine the minimum pressure required to effectively increase the DC and mechanical properties of the thermally activated acrylic resin. Thus, laboratory-scale equipment capable of applying sufficiently high pressures during the polymerization must be made available.

Regarding the post-pressing time, Consani et al. [23] reported that longer periods promote a lower percentage of residual monomer because of the longer residence time of the resin before being subjected to polymerization. However, our findings differ from these results. A post-pressing time of 12 h (for cycles CC12h, CCP12h, and CL12h) led to statistically lower values of monomeric conversion than shorter post-pressing times. A possible explanation for this could be that any residual monomers would no longer be present in the reaction medium after the 12 h, that is to say, monomers with reaction potential may not have participated in the polymerization due to delay in the start of the reaction. This result directly influences the manufacture of prosthesis, in relation to the logistics of dental laboratories, and has a clinical impact; if the waiting time for polymerization can be decreased, the prosthesis can consequently be installed more quickly.

Longer polymerization cycles were previously found to favor the production of high-molecular-weight polymer chains, improving the hardness and preventing porosity due to the lower amount of residual monomers [16]. However, in our present study, the short cycle led to similar and in some cases even statistically increased DC (Table 2). This may be explained by the fact that the short cycle included a longer period of final boiling at 100°C (1 h) compared with the long cycle (30 min). Longer final boiling is known to be a factor favoring increased conversion of monomers to polymers [17, 24]. Nisar et al. [17] showed that groups that were kept at 100°C for at least 30 min showed less residual monomer content when compared to those that did not undergo this process. These authors obtained the best results for samples that remained at 100°C for longer times, that is, a long heat treatment promoted cross-linking of the polymer chains [5]. Thus, our short cycle that included a longer final boiling period of 1 h was more favorable than our long cycle that included a shorter final boiling period of 30 min. The post-pressing time has an influence on the optimization of the prosthesis surface. A short cycle helps reduce the delivery time of the prosthesis, which is clinically relevant, mainly for hospitalized patients who require installation of internal prostheses, as it reduces the risk of infection.

The polymerization of PMMA is an addition reaction in which, theoretically, no by-products are formed [25, 26]. However, while the DCS of >90% for the twelve groups of specimen analyzed were in accordance with typical results found in the literature [17, 27–29], they are less than 100% and, hence, indicate the presence of residual monomers, as shown in Tables 3–6. This can probably be ascribed to the inhibition of free radicals that are responsible for breaking the carbon–carbon double bonds by reacting with substances other than MMA [22]. The prepolymerized powder used in the preparation of PMMA dentures contains pigments, fibers, and, especially in case of the resin used (Lucitone 199), a higher percentage of plasticizers that improve the impact strength. These additives could react with the free radicals and, thus, be responsible for the presence of residual monomers. The influence of these components is reflected by shifts of the MMA-related FTIR bands when compared to the PMMA-related bands (Figure 2) and is expected because the distinct chemical environments of each molecule promote specific vibrations.

We must note that this work only takes into account the degree of conversion of PMMA. While there is a link between the DC and the biocompatibility as well as the mechanics of this material, the present study should be complemented by cytotoxicity tests as well as measurements of mechanical and physical properties such as impact strength, hardness, surface roughness, and porosity. Only a full evaluation of the material will allow the determination of the best polymerization cycle or the best combinations of variables for the processing of PMMA-based dentures.

5. Conclusion

The post-pressing time and application of pressure did not influence the degree of conversion of PMMA. However, these factors may influence other PMMA characteristics such as porosity and mechanical properties, and additional investigative work is required.

Data Availability

The data referring to the degree of conversion values of each sample used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this article.

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