The Effect of Disinfection Techniques on the Flexural Strength of Thermopolymerisable Acrylic Resins With or Without Pigment Addition

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Authors

Marcelo Coelho Goiato *
(DDS, MSc, PhD)

Bruna Carolina Rossatti Zuccolotti
(DDS)

Marcela Filié Haddad
(DDS, MSc, PhD)

Amália Moreno
(DDS, MS, PhD)

Aldiéris Alves Pesqueira
(DDS, MS, PhD)

Humberto Gennari Filho
(DDS, MSc, PhD)

Daniela Micheline dos Santos
(DDS, MSc, PhD)

Address for Correspondence

Marcelo Coelho Goiato *

Email: goiato@foa.unesp.br

Phone: (18) 3636-3246

* Department of Dental Materials and Prosthodontics, Araçatuba Dental School, São Paulo State University, Araçatuba, SP, Brazil, 16.105-050

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ABSTRACT

Objectives: The aim of this study was to assess the flexural strength of two brands of thermopolymerisable acrylic resins (Onda Cryl, Artigos Odontológicos Clássico Ltda, São Paulo, SP, Brazil; and Lucitone 550, Dentsply, York, PA, USA) with varying concentrations of pigment (Poli-Côr, Artigos Odontológicos Clássico Ltda, São Paulo,SP, Brazil) under the influence of thermocycling, storage and disinfection.

Material and Methods: A total of 210 samples were manufactured (105 for each acrylic resin brand), with dimensions of 64 x 10 x 3.3 mm. The samples were divided into 30 subgroups (n=7) according to the proportion of pigment used (without pigment, 3% and 7%), the assessment period (initial or thermocycling for 2000 cycles) and disinfection method (immersion in 1% sodium hypochlorite, (Apothicário, Araçatuba, SP, Brazil), microwave energy or immersion in alkaline peroxide (Efferdent, Pfizer, Morris Plains, NJ, USA). The samples were submitted to the flexural strength test before and after thermocycling, and after storage with disinfection. The disinfection process was performed every 3 days, for 60 days. Data were submitted to analysis of variance (ANOVA) and Tukey test ($p<0.05$). Results: The factors that provided statistical alteration in flexural strength values were resin type and assessment period. The Onda Cryl resin and the period after disinfection (126 ± 25 MPa) exhibited the higher values of flexural strength. Conclusion: Following disinfection, Onda-Cryl resin exhibited the highest values of flexural strength. All the samples obtained are considered clinically acceptable.

INTRODUCTION

Patients rehabilitated with total prostheses aim to restore aesthetics and lost functions. Therefore, acrylic resins with different colors were developed, as well as pigments and opacifiers nanoparticles that can be incorporated to the resins[17] in order to provide a natural appearance to these prostheses. As life expectancy of the world population has increased, it is important that the materials used for prostheses present suitable mechanical strength. Since the ageing population is generally affected by systemic pathologies and the use of a range of medications that may reduce their manual dexterity the risk of dropping the prosthesis, at the time of insertion, removal or cleaning is increased, which may ultimately cause fracture.[2]

Besides, because of the contact of the prostheses with the oral cavity fluids, as well as its constant cleaning and handling by the patients, the acrylic resins may acquire an irregular surface of scratches, cracks, fractures and color change,[6,9,10,22] which may reduce the lifetime of the prostheses.[18]

The disinfection of total prostheses may be mechanical, chemical[13,24] or a combination of both techniques,[23] and it may also be performed by microwave energy.[7,8] Previous research demonstrates that frequent exposure to disinfectants may interfere with physical properties of the acrylic resins;[28] therefore, the choice method for disinfection is critical and must be based on antimicrobial properties and compatibility with the material.

It is possible to assess the physical and mechanical properties of acrylic resins by submitting them to *in vitro* tests that attempt to stimulate clinical conditions.[15] Among the methodologies developed for this purpose, thermocycling and accelerated aging,[22] cycles of disinfection and storage,[12,23] and flexural strength test,[7,11,15,18] are most relevant here.

Thus, this study aims to assess the effect of thermocycling and different disinfection techniques on the flexural strength of two brands of thermopolymerisable acrylic resins with or without the addition of pigments. The null hypotheses are 1) there will be no difference among the values of flexural strength between products, 2) the addition of pigments will result in higher values, 3) the flexural strength will decrease after thermocycling and the disinfection period, and 4) there will be no difference among the disinfection techniques applied.

MATERIALS AND METHODS

Two brands of medium pink thermopolymerisable acrylic resins were assessed (Onda Cryl, Artigos Odontológicos Clássico Ltda, São Paulo, SP, Brazil; and Lucitone 550, Dentsply, York, PA, USA).

To obtain the samples, a metal pattern with dimensions of 64 X 10 X 33mm[11,15] was placed into a Teflon metallic muffle, using type IV stone plaster (Durone, Dentsply, York, PA, USA) and extra hard laboratorial silicone (Zhermack, Badia Polesine, RO, Italy).

After silicone polymerization and plaster crystallization, the metal pattern was removed from the muffle, in order to obtain a mold for the acrylic resins pressing.

Then, the resins were proportioned and manipulated in accordance with the manufacturer´s instructions, with or without addition of specific purple pigment used on the characterization of denture base acrylic resin (Poli-Côr, Artigos Odontológicos Clássico Ltda, São Paulo, SP, Brazil), in a percentage of 3% or 7% in relation to the resin powder, proportioned through weighing in a precision digital scale accurate to 0.01 g (BEL Equipamentos Analtico, Piracicaba, SP, Brazil).

The resins were polymerized according to the manufacturer's instructions. Thus, for Lucitone resin polymerization, the muffle was kept in water at a temperature of 73ºC for 90 minutes and then, maintained in boiling for 30 minutes. For Onda-Cryl resin, the polymerization was carried out by placing the muffle at the center of the microwave oven plate (Brastemp, São Bernardo do Campo) set at 1.4 kW power, which was programmed for three cure stages - 3 minutes at a power of 30%, 4 minutes at 0%, and 3 minutes at 60%.

After polymerization, the samples were deflasked and finished with a maxi-cut abrasive drill (Vicking, São Paulo, SP, Brazil) in order to remove the excesses flash. Subsequently, polishing was performed by using a sequence of four water sandpapers (320, 600, 800 e 1200 grit) in an APL-4 polishing machine (Arotec, Cotia, SP, Brazil), for one minute for each grit.

A total of 210 samples were obtained and divided into 2 groups (n=105), according to the brand of resin used; and 30 subgroups (n=7), according to the quantity of pigment, the conditioning method and disinfection technique used *(Table 1)*.

After polishing, all the samples were stored in distilled water in a bacteriological incubator 37±2°C (CIENLAB Equipamentos Científicos Ltda, Campinas, SP, Brazil), for 24 hours,[12,14] to allow for further post-process polymerization.

Then, 42 samples underwent flexural strength test (initial period). For this, the samples were placed in a metallic holder with a distance of 50 mm between their margins, with a 100 N load cell in an Universal Testing Machine (EMIC, São José dos Pinhais, SP, Brazil), with a constant speed of 5mm/min. Constant load was applied following the three-point technique,[2,7,18,29] until fracture occurred.

The flexural strength for each specimen was determined using Equation 1:

$$FS = \frac{3.Fmax.l}{2.w.h^2} \quad FS = \frac{3\,\mathrm{Fmax}}{2\mathrm{wh}^2} \qquad \text{Equation 1}$$

where Fmax is the maximum force recorded (N), l is the distance between the supports (mm), w is the width of the specimens (mm), and h is the height of the specimens.

The other 168 samples were submitted to a thermocycling machine (Model MSCT-3, Convel, Araçatuba, SP, Brazil). For this test, the samples were immersed in distilled water, undergoing alternate cycles for 60 s dwell time at a temperature of 5±1°C and 55±1°C25, in a total of 2000 cycles, simulating two years of clinical use of the assessed resins. At the end of the procedure, 42 samples were submitted again to the flexural strength tests and the other 126 samples were stored in distilled water in hermetically sealed containers, in a digital bacteriological incubator (CIENLAB Equipamentos Científicos Ltda, Campinas, SP, Brazil), at a temperature of 37±2°C, for 60 days.

Table 1. Results of four-way Nested ANOVA for flexural strength.

Variation Factors	Df	SS	MS	F	P Value
Resin	1	7078	7078,4	15,7075	<0,001*
Pigmentation	2	1111	555,6	1,2329	0,293
Total Period	2	21347	10673,3	23,6847	<0,001*
Resin x pigmentation	2	2326	1163,2	2,5813	0,078
Resin x total period	2	1001	500,6	1,1109	0,331
Pigmentation x total period	4	1870	467,6	1,0377	0,389
Disinfection	2	99	49,5	0,1099	0,8959
Resin x disinfection	2	1940	970,2	2,1529	0,119
Pigmentation x dsinfection	2	3177	794,2	1,7625	0,138
Resin x pigmentation x total period	4	339	84,8	0,1883	0,944
Resin x pigmentation x disinfection	4	2801	700,3	1,554	0,188
Error	180	81115	450,6		
Total	207	124204			

*P < 0,05 denotes statistical significant difference

The disinfection of the samples was performed in every 3 days during the storage period (60 days).[13] Solution of 1% sodium hypochlorite[24] (Aphoticario, Araçatuba, SP, Brazil), effervescent alkaline peroxide tablets[13] (Efferdent, Pfizer Consumer Healthcare, Morris Plains, NJ, USA) and microwave energy were used for disinfection.[7,8]

The samples disinfected with sodium hypochlorite were immersed in the solution for 10 minutes and rinsed in tap water for one minute.[7,24] Samples disinfected with effervescent tablets were immersed in a container with one tablet dissolved in 250 ml of warm water for 15 minutes, followed by rinsing for 1 minute with water.[13] And the samples disinfected by microwave energy were immersed in a container with 200 ml of water, placed at the center of the microwave oven plate, and submitted to irradiation at a power of 650 W, for 6 minutes.[7,8,24]

After 60 days of storage and disinfection, these 126 samples were submitted to the flexural strength test, as described before.

The effects of acrylic resin type, pigmentation, period and disinfection on the values of flexural strength were evaluated with 3-way nested ANOVA. The disinfection factor was nested to the period factor because fewer than all levels of disinfection factor occur within each level of the period factor. Means of flexural strength difference were compared with the Tukey test (P<.05) *post hoc* test.

RESULTS

The factors Resin and Total Period exhibited statistically influence on the values of flexural strength (P<0.05) *(Table 1)*. Mean values of flexural strength (MPa) are presented in Table 2. By comparing the two resins, regardless the studied period and disinfection technique employed, it is noted that, in general, the Onda-Cryl resin exhibited significantly higher values of flexural strength than those exhibited by Lucitone resin *(Figure 1)*. By comparing the results of flexural strength obtained at different periods, independent from the resin and method of disinfection used, it was observed that significantly higher values were exhibited after the disinfection and storage period *(Figure 2)*.

Table 2. Mean values (standard deviation) of flexural strength (MPa) of the acrylic resins.

Period	Pigmentation	Disinfection	Resin	
			Lucitone	Onda-Cryl
Initial	0%	-	76.3 (4.6)	82.9 (21.0)
	3%	-	78.3 (15.2)	78.2 (19.8)
	7%	-	75.4 (16.1)	83.4 (18.0)
After Thermocycling	0%	-	72.9 (14.0)	90.6 (18.4)
	3%	-	81.0 (23.7)	94.3 (24.7)
	7%	-	69.2 (16.6)	94.0 (25.7)
After 60 days of disinfection	0%	Efferdent	101.7 (14.5)	124.3 (21.6)
		Hypochlorite	95.3 (13.5)	99.1 (23.9)
		Microwave	91.2 (20.4)	114.0 (21.8)
	3%	Efferdent	106.6 (23.4)	88.3 (19.9)
		Hypochlorite	88.0 (18.4)	99.6 (17.4)
		Microwave	92.2 (10.2)	97.4 (14.0)
	7%	Efferdent	99.3 (20.0)	97.9 (18.6)
		Hypochlorite	99.5 (17.6)	125.7 (25.1)
		Microwave	90.1 (13.7)	121.4 (17.5)

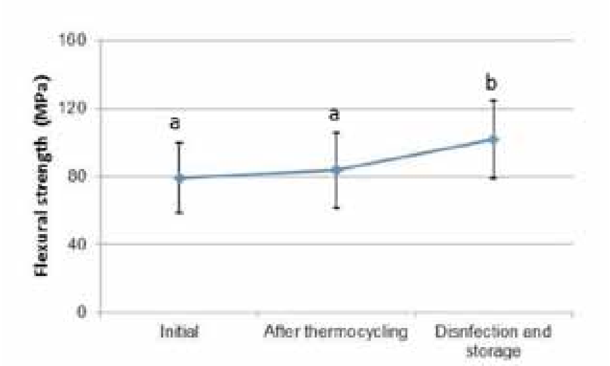

Figure 1: Mean values of flexural strength (MPa) in different resins (regardless of the period and pigmentation).

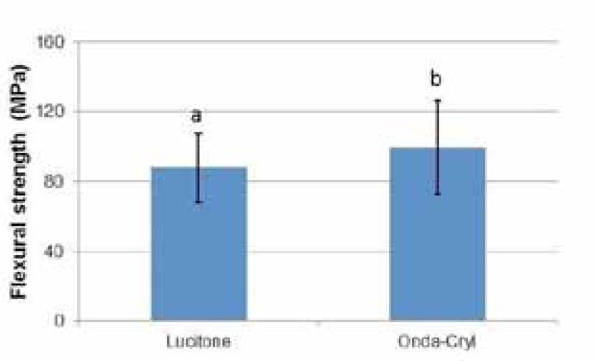

Figure 2: Mean values of flexural strength (MPa) in different periods (regardless of the resin and pigmentation).

DISCUSSION

Null hypotheses was partially rejected since there was a difference in flexural strength values between the materials after thermal cycling and disinfection.

The fracture of a denture base may be caused by several factors such as deficient adjustment, inadequate design and failures during the laboratory stages. In these situations, the denture bases are subjected to overload and it can result in fractures when the maximum mechanical capacity of the resin is reached.[15]

The most common method for measuring flexural properties of denture bases is the three-point flexural strength test, which was adopted as international standard in cases of polymeric materials, included in ISO 1567:1999 - Dentistry - Denture base polymers.[16]

Based on this methodology, Fernandes *et al.*[11] assured that the addition of pigments in acrylic resins results in greater flexural strength, due to the increase of the resin's molecular weight and generation of stronger polymeric chains.[11] However, in the current study, it was observed that only the factors "Resin" and "Total Period" significantly influenced the values of flexural strength (*Table 1*). Although TiO_2 nanoparticles are very small, facilitating its connection with polymer particles and maintaining color stability and microhardness properties, they are difficult to disperse in organic solvents and tend to agglomerate easily.[1,31,32] The agglomerations can reach a micrometer scale that are detrimental to flexural strength, incorporating porosity and increasing the likelihood of fracture.[1,31,32] Thus, these nanoparticles may require some surface modification in order to reduce the agglomeration and improve their dispersion in the resin matrix.[1,31,32]

Comparing the different brands, it is noted that the Lucitone resin exhibited lower values of flexural strength than Onda-CryI resin *(Figure 1*). It can be explained by differences in the composition and polymerization of these two materials. The manufacturers provide restricted information in the products´ leaflet, confirming only the presence of polymethylmethacrylate (PMMA) as powder base, and methylmethacrylate (MMA) as liquid base. It is known that the greater intrinsic organic content, the higher flexural strength values.[11] Thus, it is possible to assume that the Onda-Cryl resin presents such content in greater quantity.

It is known that the flexural strength is related to the efficiency of the polymerization and subsequent formation of short and low molecular weight polymer chains.[3,30] This way, it can be said that flexural strength and impact resistance of microwave resin is higher compared with the resins cured by the conventional method, what corroborates the results obtained in this study *(Table 2*).

The cycles of water bath polymerization with a terminal boil (short cycles, such as the used in the current study) significantly reduce the level of residual monomer in denture base acrylic resin.[30] The presence of residual monomer in thermopolymerisable acrylic resins subjected to long cycles increases the plasticity of the chains, as a result of an apparent transition at a temperature lower than that of the material.[30]

On the other hand, the advantage of using microwave energy is the uniform heating of the resin, since the temperature is rapidly increased and the internal and external regions are submitted to the same temperature. This uniform heating promotes strong links among the polymer chains, with low residual monomer concentration, resulting in improved mechanical properties.[11]

Regarding the analyzed periods, Figure 2 show that the flexural strength values were increased, and they were significantly higher after the disinfection period. According to the methodology applied in the current study, the effect of disinfection was added to the influence of storage in distilled water and previous thermocycling.

It is known that even after the complete polymerization cycle, residual monomer remains in acrylic resin and continues to polymerize[4,20] for approximately 14 days after mixing.[4,20,26] This post-polymerization process, especially in aqueous environments, can cause degradation of acrylic resin surface and subsurface,[20] creating regions of weakness or microdefects.[20] However, such degradation can be minimized by the presence of (EGDMA) crosslinking agents, which confer a high intermolecular resistance to materials since they form a complex of long and entwined polymer chains that reduce the space of interstitial matrix and impede water penetration.[27] These two factors together may explain the increase in flexural strength values of acrylic resins after thermal cycling and disinfection periods.

It might be expected that the water sorption would affect the polymer resistance, acting as a plasticizer and reducing the mechanical properties of material[2,5] by decreasing the forces among the polymer chains. However, the effect of mechanical property alterations, represented by the reduction on the flexural strength values, was observed only in Lucitone acrylic resin without pigment and with 7% of pigment, after the thermocycling period *(Table 2*). For the other periods of the test, it was observed that the greater the period associated with high temperatures, the greater the flexural strength values, due to the higher polymerization degree among the polymer chains.

Another explanation for the low water sorption during the other periods of the test is related to the presence of crosslinking agents that minimize the liquids absorption,[19] due to a strong association between the load and the polymeric molecule of the material, avoiding the development of porosity or cracks where the water could penetrate, and which consequently resulted in an increased flexural strength.

The standard 1567, from the International Organization for Standardization (ISO 1567, 1999)[16] indicates that the flexural strength for acrylic resins, either conventionally activated by heat or microwave-cured, must not be below 65 MPa. In the present study, the flexural strength values exceed that expectation *(Table 2*), indicating that all the groups assessed are clinically satisfactory regarding the properties investigated here.

CONCLUSION

Within the limitations of an *in vitro* study, it can be concluded that:

Onda-Cryl resin exhibited, after disinfection the higher values of flexural strength (125 +/- 79 MPa).

All the samples obtained were considered clinically acceptable in terms of mechanical strength.

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CONFLICT OF INTEREST STATEMENT

No conflicts of interest are declared for this work.

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