EVALUATION OF SOME MECHANICAL AND PHYSICAL PROPERTIES OF DIFFERENT TYPES OF INJECTABLE POLYMER MATERIALS USED AS A BASE FOR REMOVABLE ORTHODONTIC APPLIANCES

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ABSTRACT

Introduction: For many years, cold cure acrylic has been used to construct the removable orthodontic appliances that treated simple cases. The main problem with this type of acrylic is related to monomer release unlike the heat cure acrylic type. With the advances in materials, new injectable polymer materials were developed for construction of removable and complete dentures, but no study has been performed to compare their properties with Orthocryl used for removable orthodontic appliance construction.

Objectives: Aim of the study was to evaluate some mechanical and physical properties of two new materials for use in denture base construction in comparison with Orthocryl.

Material and methods: A total of 150 specimens, 50 each of self-cure acrylic (Orthocryl), polycarbonate (M10) and injectable acrylic (Acrilato), were fabricated according to the manufacturers' instructions. The following properties were tested using 10 samples of each type: impact strength, hardness, surface roughness, flexural strength, and water sorption and solubility. One-way ANOVA followed by post-hoc Tukey tests compared results among the groups.

Results: Statistically significant group differences were found in all tests except those for water sorption and solubility, which showed non-significant differences.

Conclusions: The good properties of M10 and Acrilato make them suitable alternatives to Orthocryl. The major issue is the cost and the availability of the required softening device in dental laboratories.

Key words: cold-cure, polycarbonate, injectable acrylic, orthodontics.

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INTRODUCTION

A removable orthodontic appliance consists of four main components: active, retentive, base plate and an imaginary component called the anchorage [1]. The base plate is the most bulky part of a removable orthodontic appliance, and its design varies by appliance type. It connects the components of the appliance together into a single functional unit, and provides anchorage through carrying the anchorage clasps and through contacting the mucosa of the palatal vault. It can accept many modifications such as in the posterior and anterior bite plane to treat cross bite and deep bite cases [2].

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Base plates are often made of polymethylmethacrylate (PMMA), which has been used for denture construction instead of vulcanite since the 1930s. It forms acrylic resins of three main types that are now used for the base plate fabrication: heat-cured, light-cured and cold-cured or so-called self-cure or auto-polymerizing acrylic [3].

Heat curing PMMA enhances its degree of polymerization and improves its properties, but is technically a more difficult process, so the majority of orthodontic appliances are fabricated from self-cured acrylic. Heat-cured acrylic is reserved for appliances requiring further strength [4].

Injectable polymer has recently been used to construct complete and partial dentures. Although it is flexible, it can tolerate the force of mastication. Its fabrication is also easy, clean and precise.

OBJECTIVES

Aim of the study was to evaluate the specific mechanical and physical properties of two types of injectable polymer in comparison with a self-cured acrylic resin.

MATERIAL AND METHODS

MATERIALS

Three brands of denture base materials were used: Orthocryl (Dentaurum, Germany), Deflex Acrilato FD (Nuxen SRL, Argentina) and Deflex M10 XR (Nuxen SRL, Argentina). Ten samples of each were made for each of five tests, making 150 specimens, 50 of each material, in total.

METHODS

Orthocryl was prepared according to the adapting technique. Powder and monomer liquid (2.5 parts powder to 1 part liquid) were mixed in a clean porcelain jar. Upon reaching the consistency of dough, it was quickly removed, adapted on to the model of each test and manually molded quickly to the desired shape. Curing was completed in a pressure pot at 2.2 bar, 40°C for 15 min.

The two other materials were prepared by injection according to the manufacturers' instructions. The material's cartridges were placed in an automatic programmable device (Deflex MAD) (Deflex, Argentina) and injected into the flask for 15 min under 5-7 bar at 305°C ± 10°C for M10 material and 265°C ± 10°C for Acrilato.

A suitable cartridge for the injection material was selected, and its end was sealed with a Vaseline base lubricant. The cartridge was then introduced into one of the two heating cylinders. It was oriented toward the flask chamber. The excess lubricant on the heating cylinder's margin was removed using absorbent paper. Preheating was conducted for a set time marked by an audible signal.

The flask's two halves were assembled, and fastened with screws. The flask was then placed and secured in the injecting unit. Its opening was aligned with the cartridge and the heating cylinder. Pressing the start key on the control panel then initiated the injection procedure, which took about 0.25-0.35 s. Contraction during setting was compensated by automatically keeping the pressure constant for 1 min. The cylinder was then moved about 3 to 4 mm away from the flask so that the cartridge could be separated. The flask was then removed, and the used cartridge was automatically released by pressing the evacuation key. In order to optimize the quality of the material, the flask was allowed to cool slowly for about 8 to 9 h. After cooling, the screws were loosened and the flask halves were slowly opened. The specimen was removed from the molds.

The specimens were then finished and polished except for those used in surface roughness tests. The Acrilato and M10 specimens were finished using special finishing plastic burs, while the Orthocryl specimens were finished using acrylic burs to remove acrylic flashes. All the specimens were then finished (for 15 s) using sandpaper (120 μm grain size); to avoid overheating, the specimens were cooled by immersion (for 15 s) in a rubber bowel filled with water.

Specimens were polished in a lathe polishing machine using a bristle brush and rag-wheel with pumice. A gloss surface was achieved using polishing soap on a dental lathe at low speed (1500 rpm) with continuous water cooling to avoid overheating.

MECHANICAL AND PHYSICAL TESTS

IMPACT STRENGTH

Bar-shaped specimens of 80 mm × 10 mm × 4 mm were prepared [5] and stored in distilled water at 37°C for 48 h before testing [7] using a Charpy-type impact testing device. During testing the specimens were supported horizontally at each end and struck by a free swinging pendulum with an impact energy of 2 J. The polycarbonate and injectable acrylic were tested at 30 J, because they withstood testing at 2 J. The scale readings gave the impact energy in joules. The Charpy impact strength of unnotched specimens was calculated in kJ m^{-2} using the following equation.

Impact strength = $(E/b \times d) \times 10^3$,

where *E* is the impact energy (J), and *b* and *d* are respectively the width and depth of the specimens (mm).

FLEXURAL STRENGTH

Bar-shaped specimens (65 mm × 10 mm × 2.5 ± 0.1 mm, length, width and thickness respectively) were prepared and stored in water at 37°C for 7 days before testing [6]. A universal Instron testing machine tested specimens positioned on the bending fixture, which was composed of two parallel supports 50 mm apart, with a 50 kg load (full scale). The load was applied with a crosshead speed of 5 mm/min by a centrally placed rod between the supports, deflecting the sample until fracture.

$S = 3PI/2bd^2$,

where S is the flexural strength (MPa), P is the load at fracture (N), I is the distance between the supporting wedges (50 mm). As above, b and d are the specimen's width and thickness (mm).

SURFACE HARDNESS

Specimens with dimensions of 65 mm x 10 mm × 2.5 ± 0.1 mm were stored in distilled water at 37°C for 48 h [7]. They were then tested using a Shore D durometer hardness tester, which is suitable for acrylic resins and consists of a spring-loaded indenter (1.40 mm diameter). The indenter was attached to a digital scale graduated from 0 to 100 units.

Testing involved pressing down firmly and quickly on the indenter and recording the reading. Each specimen was indented three times (once in the center and once at each end), and the mean of the three readings was calculated.

SURFACE ROUGHNESS

Bar-shaped specimens (65 mm × 10 mm × 2.5 ± 0.1 mm) were stored in distilled water at 37°C for 48 h before being tested [7] using a profilometer device with 0.001 μm accuracy. This device includes a sharp diamond stylus surface analyzer to trace the profile of surface irregularities by recording all the peaks and recesses. It characterizes the surface by its scale.

The specimen was placed on the device's stable stage, and the location of the tested area was selected (the specimen was divided into three parts). The analyzer then traversed along each tested area, and the mean of the three readings was calculated in μm.

WATER SORPTION AND SOLUBILITY

Acrylic disc specimens (50 mm ± 1 mm diameter and 0.5 mm ± 0.05 mm thick) were prepared using a metal pattern [7]. They were dried for 24 h in a desiccator containing freshly dried silica gel; the desiccator was held at 37°C ± 2°C in an incubator. Upon removal, specimens were maintained at room temperature for 1 h before being weighed with a digital balance with an accuracy of 0.0001 g. Drying and weighing cycles were repeated until a constant mass (M1, conditioned mass) was reached, which indicated that the weight loss from each disc was not more than 0.5 mg in 24 h [7].

All sample discs reached M1 after 4 days of drying, and were then immersed in distilled water for 7 days at 37°C ± 2°C [8]. Upon removal from the water with dental tweezers, they were wiped with a clean dry towel until free from visible moisture. Their mass recorded 1 min after removal from the water was labeled M2.

Water sorption (WSP, mg/cm^2) was calculated as follows for each disc [7]:

$WSP = (M2 - M1)/S$

where $M1$ and $M2$ are the disc's masses as defined above (mg) and S is its surface area (cm^2).

Solubility was measured by again reconditioning the discs to constant mass in the desiccators as described above. The reconditioned mass was recorded as M3. All samples reached M3 within 4 days of drying.

The solubility (WSL, mg/cm^2) was determined for each disc as follows:

$WSL = (M1 - M3)/S$,

where $M3$ is the reconditioned mass (mg), and $M1$ and S are as given above.

STATISTICAL ANALYSES

Data were collected and analyzed using SPSS (v. 25). The descriptive statistics included means, standard deviations, minimum and maximum values, and the inferential statistics included one-way ANOVA testing followed by Tukey's HSD testing. The probability value was set at 0.05.

RESULTS

Table 1 lists descriptive statistics regarding the materials' properties and group differences.

The mean impact and flexural strengths were ranked from highest to lowest in the order M10, Acrilato, Orthocryl, with highly significant differences among them. Tukey's HSD test revealed significant differences among the groups.

Surface roughness testing showed Orthocryl as the roughest, then M10, and Acrilato as smoothest, with highly significant group differences. The roughness difference between M10 and Acrilato was not significant, but their results were significantly different from those for Orthocryl.

Orthocryl had significantly higher surface hardness than Acrilato and M10. According to Tukey's test, the differences between Acrilato and M10 and between Acrilato and Orthocryl were not significant, while those between M10 and Orthocryl were significant. Orthocryl showed the highest mean water sorption and solubility of the three materials, but with non-significant differences.

DISCUSSION

Self-cured acrylic has long been used in the fabrication of base plates for removable appliances. It has many

TABLE 1. Descriptive statistics and group differences for the studied materials' properties

Tests	Groups	Descriptive statistics				Comparison			
						ANOVA test		Tukey's HSD test	
		Mean	SD	Min.	Max.	F-test	*p*-value	Groups	*p*-value
Impact strength (kJ/m^2)	I	39.102	4.139	32.938	42.849	1352.831	0.000	I-II	0.000
	II	208.846	10.555	195.99	218.3			I-III	0.000
	III	7.838	1.086	6.04	8.706			II-III	0.000
Flexural strength (MPa)	I	160.600	3.782	155	164	41.826	0.000	I-II	0.018
	II	173.200	6.723	165	181			I-III	0.000
	III	138.200	7.294	132	148			II-III	0.000
Roughness (µm)	I	1.765	0.268	1.406	2.058	30.831	0.000	I-II	0.745
	II	1.966	0.551	1.291	2.655			I-III	0.000
	III	3.702	0.420	3.424	4.446			II-III	0.000
Surface hardness	I	97.044	3.984	90.63	100.40	5.091	0.025	I-II	0.423
	II	93.344	1.652	91.53	95.23			I-III	0.188
	III	102.386	6.503	95.33	110			II-III	0.020
Water sorption (mg/cm^2)	I	0.005	0.002	0.003	0.007	0.831	0.459		
	II	0.005	0.005	0	0.011				
	III	0.008	0.005	0	0.014				
Water solubility (mg/cm^2)	I	–0.001	0.003	–0.004	0.004	1.228	0.327		
	II	–0.003	0.003	–0.009	0				
	III	–0.005	0.005	–0.010	0.002				

I – Acrilato, II – M10, III – Orthocryl

advantages including dimensional stability, ease of deflasking, and quick processing. On the other hand, it has disadvantages such as high residual monomer content, high creep rates, reduced stiffness, lower fatigue strength, color instability and high solubility. Orthocryl is cold-cured acrylic produced by Dentaurum; it comprises a polymer with large particles that prevent dripping of the acrylic and monomer. It has a longer working time and shorter curing cycle below 40°C [9, 10].

This study aimed to evaluate some physical and mechanical properties of two materials currently used in the construction of partial dentures (namely M10 and Acrilato) in comparison with Orthocryl.

Fourier transform infrared spectroscopy has shown the major component of the extra-rigid polymer M10 to be polycarbonate and that of Acrilato injectable acrylic to be PMMA [11].

IMPACT STRENGTH TESTING

Table 1 shows that M10 possessed the highest mean impact strength, followed by Acrilato and then Orthocryl, with a statistically significant difference. These results can be explained from different aspects. First consider differences in the materials' chemical structures. The high impact strength of M10 is related to the high aromatic content (phenyl groups, benzene rings) of its backbone and the moderately large amount of pendent oxygen and hydrogen groups. The latter groups facilitate tangling, with the polymer chains approaching close to each other to form hydrogen bonds. These aspects will enhance the material's resistance to intermolecular movements [12]. On the other hand, M10 has a high capacity for plastic deformation without cracking or breaking, unlike PMMA [13]; moreover, M10 samples are ductile at room temperature and can be bent without fracturing when force is applied [14].

PMMA is a thermoplastic, amorphous polymer, and its mechanical properties are affected by the testing temperature. Its behavior will change from brittle to ductile as it is heated close to its glass transition temperature (average 104°C). It has a limited range of working temperatures, and undergoes brittle fracture at room temperature. It is unlike M10 (glass transition temperature 145°C), which has the unique property of being less affected by temperature and able to maintain its mechanical properties under a wide range of temperatures [10].

Next consider the materials' different processing techniques. The higher impact strength of injectable PMMA (Acrilato) was attributed to its dual polymerization, which resulted in the end polymer containing less (or no) free monomer than Orthocryl, whose reduced impact strength is related to its residual monomers and under-polymerized chains [15].

FLEXURAL STRENGTH TESTING

The mean flexural strength results were ranked from M10 with the highest, to Acrilato, and then Orthocryl with the lowest, because injection-molded polymers are more flexible and ductile [16, 17]. The differences between M10 and Acrilato were attributed to differences in their chemical structures influencing their mechanical properties. M10 is produced by the linear polymerization of bisphenol A (BPA) and phosgene $COCl_2$, which react in more than one molecular space to form a three-dimensional cross-linked network. The structure builds up by a condensation reaction, and provides M10 with superior flexibility and elasticity at room temperature; however, Orthocryl is formed by free-radical polymerization of the monomer (assisted by a catalyst), which reacts in one molecular space to form a two-dimensional network that increases in size by an addition reaction, resulting in a brittle polymer at room temperature [18].

An alternative explanation of the differences between Orthocryl and Acrilato is related to their different polymer families. Impact strength is inversely related to rigidity, and changes of the polymer properties are related to the modifications of its chemical composition that are necessary to facilitate a certain processing technique [13].

SURFACE ROUGHNESS TESTING

Orthocryl showed a significantly rougher surface than M10 and Acrilato, and it contained larger polymer particles than the others. Another potential factor affecting the surface roughness is the type of initiator in the acrylic resin system [9], because the high residual monomer content in Orthocryl is related to the low degree of polymerization achieved by the chemical activator. A further possible reason is the structure of Orthocryl being more porous than the others [19, 20]. The amount of pressure used during curing is also an influence, as it reduces the porosity, leading to a smooth surface. M10 and Acrilato were nearly monomer-free (being processed at 5-7 bar pressure), and showed regular, non-porous structures.

HARDNESS TESTING

Orthocryl showed the highest mean hardness, followed by Acrilato, and then M10 (polycarbonate).

Both Orthocryl and Acrilato have PMMA chemical structures, and the difference in their processing techniques has previously been shown not to significantly affect their hardness [9]. This supported the finding of the present study, in which no significant difference was found between Orthocryl and Acrilato.

A significant difference between M10 and Orthocryl was observed because of the differences in their molecular structures, which affected the molecular interaction forces. Frank energy and friction force related to the depth of indentation also affected polymer hardness.

When a polymer is indented by a durometer tip the area under the indenter will compress owing to the force on the polymer's surface, displacing the polymer chains and packing them within the elastic and plastic limit of the polymer. A polymer's resistance to indentation can be explained by the strain gradient formulation for elasto-plastic materials. This suggests that polycarbonate's aromatic rings provided increased surface hardness during initial indentation up to a depth of 20 μm, after which the hardness decreased owing to greater plastic deformation. This is in comparison with Orthocryl, whose lack of aromatic rings in its complex structure may explain the increase in hardness at small indentation depths [17].

Another possible explanation is that the chemical structure and arrangement of polymer molecules affect the bending stiffness of the polymer chain and so influence the overall Frank elasticity of the polymer [21]. The molecular structure and network arrangement of polycarbonate chains give them higher bending stiffness and greater elasticity (and less resistance to indentation), while the structure and arrangement of the PMMA chains in Orthocryl result in lower bending stiffness and greater rigidity [8, 22].

WATER SORPTION AND SOLUBILITY TESTING

Acrylic resin's water sorption and release cause dimensional instability so that subjecting the material to internal stresses will lead to cracks forming, and finally fractures [23, 24].

Water may interact with the polymer chains and spread between the macromolecules of the material, forcing them apart, leading to effects such as reversible loosening or effective plasticization of the structure, solvation or reversible rupture of weak inter-chain bonds, and irreversible disruption of the polymer matrix. This affects the material's dimensional behavior, durability and stability [25].

In the present study, Orthocryl had greater water sorption and solubility than M10 and Acrilato, but with a non-significant difference. This represents an advantage of the injectable materials over Orthocryl, as M10 consisted of polycarbonate (which lacked free monomers) and the major component of Acrilato injectable acrylic was PMMA. Various reasons account for this.

First consider the homogeneity of the material, which Miettinen *et al.* [26] stated affected the water sorption and solubility of polymers, with greater homogeneity leading to less water absorption and lower solubility [27]. High porosity will assist the transportation of fluid in and out of the network, leading to enhanced water uptake and elution [28].

The second factor is free monomer. Initiators, plasticizers and free monomer are soluble materials present in acrylic resins. A positive correlation between the amount of residual monomer and weight loss during solubility tests has been suggested [29]. Therefore, the first few days of water storage usually see the largest amount of residual monomer leached from acrylate. M10 was monomer-free, and Acrilato contained little monomer, in contrast to the large amount of residual monomer in Orthocryl.

The chemical nature of the polymer is a third consideration. Acrylic resins absorb water gradually over time principally because of the polar properties of the resin molecules. A high equilibrium uptake of water can soften the resins as absorbed water can plasticize acrylate and reduce the material's strength [29].

The extent and rate of water uptake into polymer networks are predominantly controlled by resin polarity – which is dictated by the concentration of polar sites available to form hydrogen bonds with water – and network topology [28, 30].

Arima *et al.* [31] stated that the relation of the chemical nature of a polymer to that of water directly affects water sorption by the resin. Water is absorbed into a polymer with polar molecules containing unsaturated bonds or with unbalanced intermolecular forces. Dixon *et al.* [32] reported that residual monomers can influence water sorption and expansion.

Finally, pressure during flasking or pressure applied during processing can prevent the boiling of the monomer. Therefore, curing under pressure can prevent the formation of porosity, making increased curing pressure appear to be helpful in decreasing the porosity of acrylic resins [19, 20]. Monomer vaporation associated with the exothermic polymerization reaction and inadequate pressure may cause porosity in resin specimens [33]. Water is absorbed into voids in the resin [26], so specimens cured at low pressure (such as Orthocryl) have greater water sorption and solubility. This agrees with the finding of Miettinen *et al.* [34] in that polymerization pressure influenced water sorption, with higher pressure leading to lower water sorption.

CONCLUSIONS

This *in-vitro* study indicates that M10 and Acrilato are suitable alternatives to Orthocryl as materials for orthodontic base plates. They showed many advantages over Orthocryl, being quick and easy to polish and ductile, while possessing high impact and flexural strengths, and showing low roughness, water sorption and solubility, and comparable hardness. The major issue is the cost and availability of the required Deflex MAD device in dental laboratories.

Future research is necessary to investigate other physical properties of these materials. The adhesion of bacteria and *Candida* to their surfaces and the effect of denture cleanser on their properties are also important further factors to be studied.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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