Original Article

Analysis of the Mechanical Behavior and Surface Rugosity of Different Dental Die Materials

Ciro T. Niekawa, Simone Kreve, Gisseli Bertozzi A'vila, Gilmar Gil Godoy, Eduardo Vieira da Silva J. R., Sergio Candido Dias

Department of Restorative Dentistry, São Leopoldo Mandic Dental School, Campinas-São Paulo, Brazil

Abstract

Aim: This work evaluated the mechanical and surface behavior of different die materials. The studied materials are polyurethane resin Exakto-Form (Bredent), Gypsum type IV, Fuji Rock EP (Gc), and Durone (Dentsply).

Materials and Methods: Two metallic matrices molded in polyvinyl siloxane provided 30 cylindrical test specimens for the diametral compression test and 30 hemispherical test specimens for the surface rugosity test. The cylindrical test specimens were submitted to tests of diametral compression strength using a DL2000 universal assay machine, with a load cell of 2000 Kgf and constant speed of 1 mm/min connected to the software. Kruskal–Wallis and Dunn's nonparametric tests were used to analyze the results. The hemispheres were submitted to the surface rugosity assay using a SJ201-P rugosimeter with a sensitivity of 300 µm, speed of 0.5 mm/s, and cut-off of 0.8 mm, and the readings were taken on the convex surface of the test specimens and metallic matrix. Results were analyzed using with Fisher's least significant differences test (LSD) and Dunnett's test.

Results: Kruskal–Wallis test showed significant difference between die materials for diametral compression strength ($P = 0.002$). Dunn's test showed significantly higher values for modified polyurethane resin (Exakto-Form). The gypsum type IV, which did not significantly differ regarding diametral compression strength, showed 34.0% (Durone) and 42.7% (Fuji Rock) lower values in comparison to Exakto-Form.

Conclusion: Within the parameters adopted in this study, it is possible to conclude that Exakto-Form polyurethane resin showed higher resistance to compression and was closer to the metallic matrix rugosity, and, along with the gypsum type IV Durone, showed better reproducibility of details relative to the Fuji Rock.

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Introduction

Dental gypsum is widely used and studied for its use in obtaining dental casts.[1-3] Plaster and stone products used in dentistry are made by calcining calcium sulfate dihydrate. The principal constituent of gypsum-based products is calcium sulfate hemihydrate,[4] $(C_aSO_4)^{2}\cdot H_2O$. Basically, natural plaster production occurs in four steps, namely, gypsum extraction, calcination preparation, calcination, and selection. Gypsum is a sedimentary rock that is essentially composed of gypsite, anhydrite, and a few impurities, usually clay minerals, calcite, dolomite, and organic matter. Gypsite is a compact material with low hardness and low solubility in water and is the raw material of die.[5]

Popularity of type IV gypsum is attributed to its ease of use, relatively quick setting, and reasonable accuracy.[6] A crucial factor in the success of this process is having a model that is both accurate and possesses a smooth surface.[7]

In dentistry, die is very relevant owing to its use in studying and working models.[8] Dental models are subjected to constant handling, and hence, they must be fracture resistant for safe laboratorial procedures.[9] The surface properties of the die stone influence its ability to tolerate all type of forces during a restoration.[3]

All die materials present some dimensional changes on solidification or hardening. In general, hardness refers to "resistance to indentation or scratching."[10] Some present low rugosity, jeopardizing the reproducibility of the impression material; some fail to adequately reproduce the details present in impression.[7,11] Plaster type IV is the most commonly

Address for correspondence:
Ms. Simone Kreve, E-mail: simonekreve@hotmail.com

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material used in working models and as auxiliary in laboratory procedures involving dental prosthesis that comply with the American Dental Association (ADA).[12] Because Types IV and V dental stones are used for the fabrication of definitive casts, their dimensional stability is important.[13] It is, however, a material with some limitations such as low resistance to fracture and abrasion.[8,14]

Several attempts have been made to develop a die material with improved properties. Areas of interest include improved mechanical strength such as resistance to fracture, surface hardness, and resistance to abrasion, and surface improvements such as surface rugosity, accuracy, and detail reproducibility.[8,10,15]

To obtain more accurate and durable dental casts, some alternative systems have been suggested such as die metallization, synthetic die, epoxy resin,[16-18] polyurethane resin reinforced stone, and electroformed dies.[19,20]

Polyurethanesare high industrial performance polymers that, given the ample choice of reagents available, give origin to a wide range of compounds with different chemical and physical properties. Acording to Derrien and Sturtz,[18] polyurethane resins are naturally flexible and provide sufficient transverse strength. Polyurethane is a biocompatible material and in dentistry it is used in several fields of expertise,[21] such as in prosthetics and implants studies, where it replaces the bone tissue in *in-vitro* assays.[22]

Considering the need for more accurate and resistant models capable of reproducing prosthetic works with a desirable clinical behavior, this study aims to evaluate the resistance to fracture of polyurethane resin models through diametral compression comparing the results to those of die type IV, as well as to evaluate these materials ability to reproduce the details in the model using surface rugosity analysis.

Materials and Methods

The following three die materials were assessed [Chart 1]:

- Exakto-Form (Bredent, Senden, Germany) polyurethane resin
- Durone (Dentsply, Petropolis, Brazil) type IV die
- Fuji Rock (Gc Europe, Leuven, Belgium) type IV die

With the aid of the software AutoCAD, a matrix was developed in Aluminum at the precision workshop PoçostecLtda (Poços de Caldas - MG) for the test of diametral compression strength. This matrix is composed of three threaded parts [Figure 1], whose base holds four equidistant Teflon cylinders. The inferior portion of the base is articulated with the portion holding the Teflon cylinders through an M6 Allen screw, allowing the unscrewing without transmitting tension to the impression materials. The two remaining parts are the matrix tray's body and cap. The body is threaded to the base to restrain the impression material, preventing lateral shifts. This set is composed of three identical trays, allowing the production of three simultaneous impressions that were left to rest for the time specified for each material and, after pouring the impression materials, resulted in 12 cylindrically-shaped test specimens with dimensions of 12 mm (length) and 6 mm (diameter), originating from the same mixture.

For the surface rugosity test, a matrix was developed in aluminum at PoçostecLtda (Pocos de Caldas - MG, Brazil) with the aid of the software AutoCAD aiming to facilitate its reproduction in any precision workshop. This matrix is composed of two parts [Figure 2].

One aluminum tray for the impression material and another for the aluminum model composed of six equidistant cavities which, following the die materials pouring, gave origin to 6 test specimens with a flat surface of 60 mm width and a convex surface with 50 mm height produced from the same mixure.

Three trays were used for the test specimens. The trays were mounted and impression were taken one at a time, previously sealing the teflon tray periphery with LC block-out (Ultradent do Brazil - São Paulo-SP) photopolymerizable laboratory resin to facilitate the unscrewing of the tray [Figure 3]. The Impressions were then taken of the Teflon cylinders using the double mixture technique (light body/putty) with an addition-reaction silicone polymer Elite (Zermack, São Paulo, Brazil). The light body material was injected with an automixpistol (Zermack, São Paulo, Brazil) on the Teflon cylinders and on the putty previously handled and activated. Then, all the material was fit into the tray. The matrix tray was slowly screwed together to allow the impression material flow through the designed orifices, resulting in higher fidelity in the impression process. Three impressions were obtained resulting from the three trays and left to rest for 1 hour. Following that, the set was unscrewed and three molds with four cylindrical cavities (corresponding to the Teflon pins)

Chart 1: Materials used in the assays

Fuji Rock EP plaster type IV: (Gc Europe, Leuven, Belgium)	Durone plaster type IV (Dentsply, Petrópolis, RJ, Brazil)	Exakto-Form polyurethane resin (Bredent, Senden, Germany)
Alpha-hemihydrate calcium sulphate and dye	Alpha-hemihydrate calcium sulphate and dye	Components A and B
Water–powder ratio: 19 ml/100 g	Water-powder ratio: 19 ml/10 0g	Mixture proportion 1:1
Initial setting time: 12 minutes	Initial setting time: 8 minutes	Mixture time: 30 seconds
Setting expansion: 0.08%	Setting expansion: 0.09%	Working time: 2–3 minutes
Resistance to compression: 53 Mpa	Resistance to compression: 1 hour - 7000 psi/7 days - 15000 psi	Impression separation: after 30 minutes
Test methods: ISO 6873	Batch: 9797010	Maximum final polymerization: 1 hour and 30 minutes
Batch: 1404084		Batch: 20111201

were obtained [Figure 4a and b]. These impressions were filled with type IV Durone and Fuji Rock die (vacuum handled following the manufacturer's instructions, which established a certain powder-water ratio determined with digital scale and graduated syringe) and Exakto-Form polyurethane resin (handled according to the manufacturer's instructions). The flow of the model material was accomplished with the use of brushes and mechanical vibration. The polyurethane resin leakage did not require vibration owing to its high fluidity, and it was accomplished with the use of a 20 mL disposable syringe [Figure 5a and b]. After the required hardening time, the models were separated from the impressions, resulting in 10 test specimens for each material along with 2 test specimens for disposal.

The diametral compression strength assay was conducted according to ADA #25 specifications for dental dies.[12] The test specimens were placed over a flat base at the EMIC - DL2000 apparatus (São José dos Pinhais, SP, Brazil) one at a time and compressed with a 2000 kgf load cell at a constant speed of 1 mm/min until fracture. Results were registered with the software connected to EMIC - DL2000 (São José dos Pinhais, Pr, Brazil) as displacement and strength values.

The tray was mounted on a flat base for preparing the test specimens and the six hemispheres were molded using the double mixture technique with addition-reaction silicone polymer Elite (Zermack). The light body was injected with an automixpistol over the hemispheres to accommodate the dense material, which covered the entire tray. With the aid of a glass plate, the impression material was submitted to uniform pressure, leading to the leakage of excessive material through the metallic matrix lateral reliefs and mold stabilization until the final setting. After 1 hour, the aluminum mold was removed and the quality of the addition-reaction silicone polymer model was assessed. The modeling materials were leaked in the same technique described previously. Following the hardening time, the models were separated from the impressions and the same method was repeated

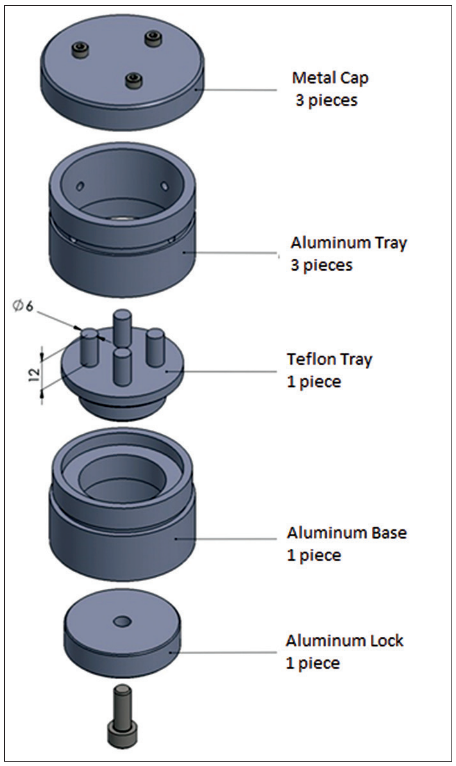


Figure 1: Design of the matrix for the Compression resistance test (sequence of fittings). Source: own elaboration

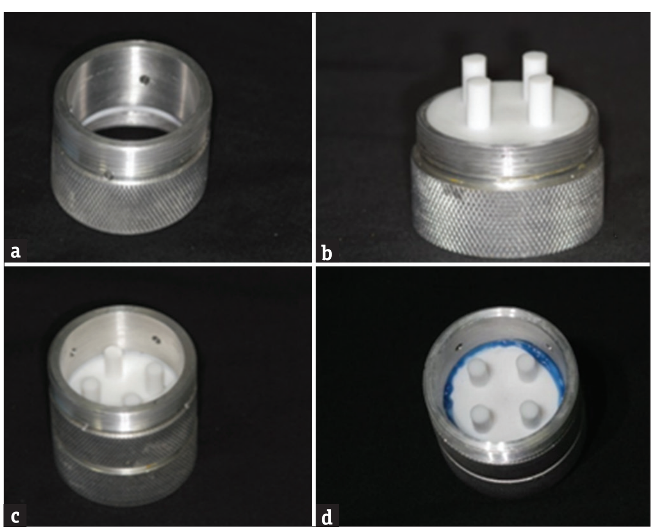


Figure 3: Sequence of the matrix mounting for the diametral compression assay Caption: (a) aluminum tray (b) base of the tray articulated to the Te on pins (c) threaded base and body (d) sealing of the Te onperiphery with lc block-out (Ultradent do Brasil-São Paulo/ SP). Source: own elaboration

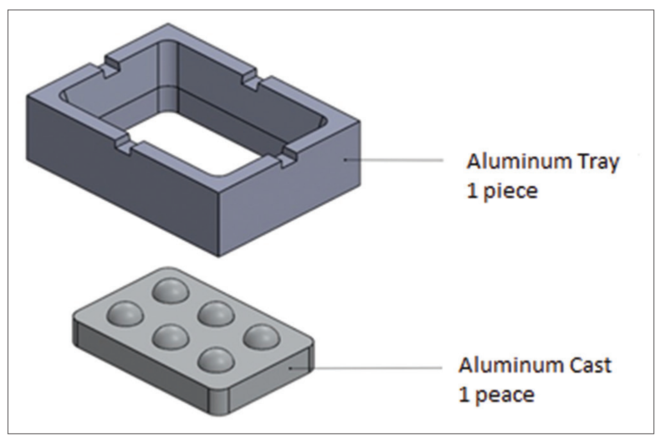


Figure 2: Design of the matrix for the surface rugosity assay Source: own elaboration

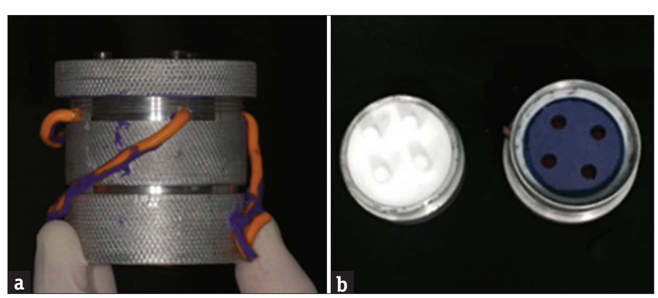


Figure 4: Matrix molding for the diametral compression assays Caption: (a) molding with polyvinyl siloxane leakage on the matrix orifices (b)mold obtained of the Teflon pins Source: own elaboration

until 10 test specimens and 2 specimens for disposal were obtained [Figures 6-8].

For the rugosity test, readings were taken at the convex surface of the test specimens [Figure 6]. The surface rugosity (Ra) was measured with a rugosimeter SJ201-P Mitutoyo (Kawasaki, Kanagawa, Japan), with a sensitivity of 300 µm, speed of 0.5 mm/s, and cut-off of 0.8 mm. This device has a pick-up motorized needle in the transversal unity that takes the readings and can be moved in the vertical axis to fit the test specimen height and in the horizontal axis to read the surface rugosity. The device also has a programmer unity that registers the readings. The test specimens and the matrix with the aluminum mold were strapped with double-sided tape to a base tilted by 14° and the readings were taken one at a time. The device was calibrated with a metallic standard provided by the manufacturer with 2.97 µm Ra. Five measurements were taken for each test specimen at a point equidistant from the center of the sample and their average was used to represent the surface rugosity.

Comparison between impression materials was done using Kruskal–Wallis and Dunn's nonparametric tests because the dataset showed variance heterogeneity that could not be stabilized by transformations.

The rugosity results obtained for the impression materials on the convex surface and for the matrix were submitted to variance analysis at one criterion. Fisher's least significant difference (LSD) and Dunnett's test were used in multiple comparisons.

Statistical analysis was done using the Statistical Package for the Social Sciences version 20 (SPSS Inc., Chicago, IL, USA), with the significance level of 5% ($\alpha = 0.05$).

Results

Table 1 shows the descriptive analysis with means and standard deviations of resistance to diametral compression and rugosity of the convex surface of plaster type IV (Duroneand Fuji-Rock) and of the modified polyurethane resin (Exakto-Form). Matrix surface rugosity is also reported.

Kruskal–Wallis test showed significant difference between the impression materials for the diametral compression strength ($P = 0.002$). Dunn's test showed significantly higher results for the modified polyurethane resin (Exakto-Form). Type IV dies, which did not significantly differ from each other in terms of diametral compression resistance, showed 34.0% (Durone) and 42.7% (Fuji Rock) lower results in comparison to Exakto-Form [Table 1 and Graph 1].

Variance analysis showed significant difference ($P < 0.001$) in surface rugosity depending on material – impression or matrix. Jointly comparing die type IV, modified polyurethane resin, and matrix, Fisher's LSD test showed that the rugosity of the convex surface of the Fuji Rock was significantly smaller than that found in all other materials (Durone, Exakto-Form resin, and matrix). The Durone showed significantly smaller rugosity than that of the modified polyurethane resin. The rugosity of the matrix was not significantly different from that of Durone or modified polyurethane resin [Table 1 and Graph 2].

Table 1: Diametral compression strengthand surface rugosity of plaster type IV, modified polyurethane resin and metallic matrix

Material	Resistance* (Kgf)	Rugosity$^{\yen}$ (µm)
Durone	104.22 (53.31) B	4.82 (0.46) B
Fuji Rock	90.49 (24.08) B	4.35 (0.50) A
Exakto-Form	157.81 (5.60) A	5.54 (0.55) C
Metallic matrix	–	5.14 (0.40) BC

*, letters indicated by Dunn's test; $^{\yen}$, letter indicated by Fisher's LSD test. Averages followed by different letters show significant differences between materials in the same column

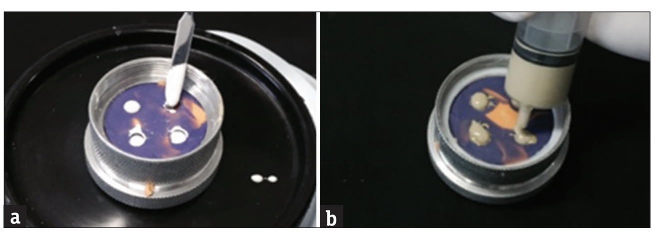


Figure 5: Modeling material leakage Caption: (a) type IVdie (b) polyurethane resin Source: own elaboration

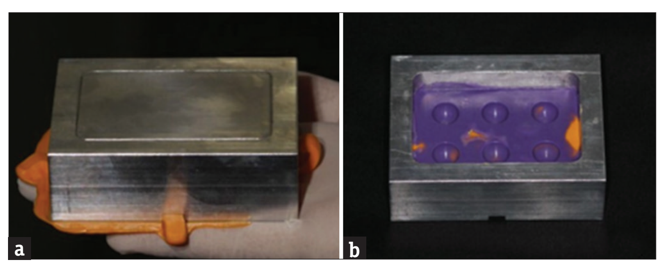


Figure 6: Matrix molding for the surface rugosity assays Caption: (a) matrix molding with polyvinyl siloxane (b) mold on the hemispheres Source: own elaboration

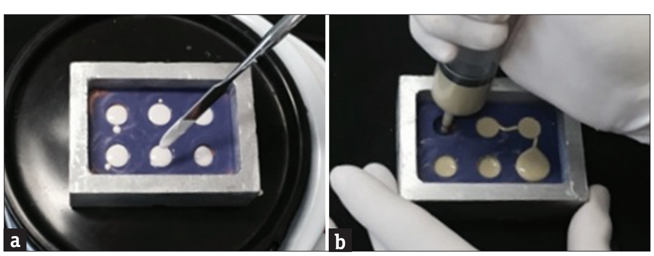


Figure 7: Modeling material leakage Caption: (a) type IV die leakage (b) polyurethane resin leakage Source: own elaboration

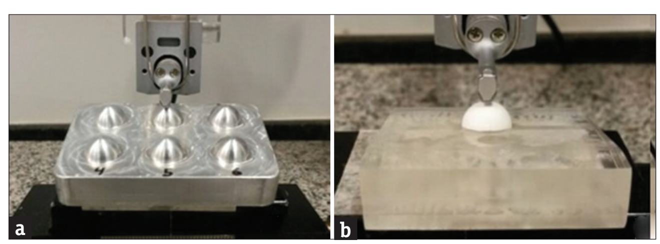
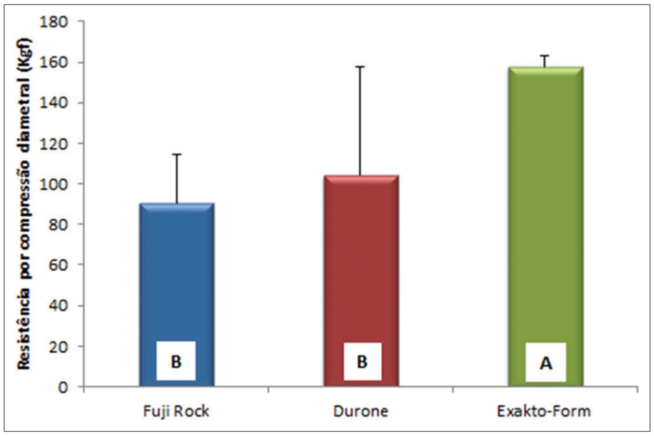
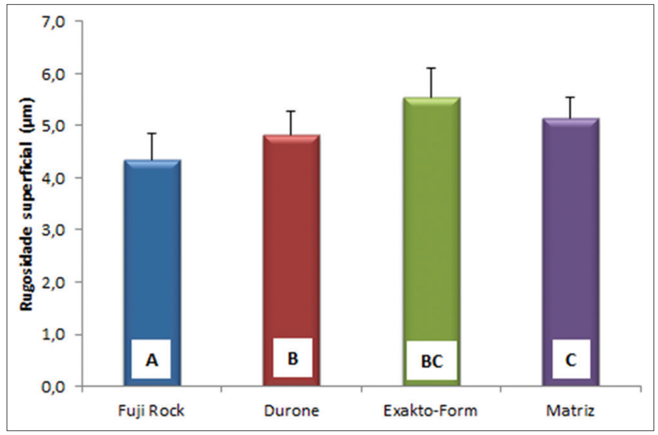


Figure 8: Reading of the convex surfaces of the matrix (a) and test specimens (b) Source: own elaboration



Graph 1: Column diagram of diametral compression strength averages of plaster type IV and modified polyurethane resin



Graph 2: Column diagram of rugosity averages of the convex surface of plaster type IV, modified polyurethane resin, and matrix

Confirming the Fisher's LSD test results, Dunnett's test showed that only Fuji Rock type IV die showed significantly lower surface rugosity lower than that found in the matrix when comparing the impression materials with the matrix. All remaining materials showed no significant difference in surface rugosity in comparison to the matrix.

Discussion

To obtain an accurate model, both impression and die materials should have positive properties.[2] The die material should be compatible with the impression material.[23] Casts poured in dental stones should be accurate in every respect, dimensionally stable over time, hard enough to withstand the fabrication process, resistant to the inadvertent abrasions caused by fabrication, and have a surface wettability compatible with the waxing process.[8]

However, gypsum casts show low tensile resistance and are prone to fractures,[23,24] with a poor reproducibility of details.[5,25] The development of synthetic resins allowed the search for alternatives to the use of dies, aiming at improved mechanical resistance and precision, such as the studies conducted by Derrien and Sturtz,[18] Dias *et al*.,[9] Gujjarlapudi *et al*.[11] Kumar and Garg,[26] Black,[21] and Lillywhite *et al*.[19]

The strong, yet flexible, properties of polyurethane may be an alternative dental arch model.[27] In one study, the dimensional changes for polyurethane resin in comparison with other die materials were statistically insignificant.[28] In addition, the desired abrasion resistance of polyurethane dies results in a dense and nonabsorbent surface, facilitating easy lift off of patterns.[19] The results, both by the Kruskal–Wallis test and Dunn's test, showed increased strength by diametrical compression for polyurethane resin Exakto-Form (Bredent, Germany) with a significant difference over the other tested materials, i.e., plaster type IV Durone (Dentsply, Brazil) and Fuji Rock (Gc Europe, Belgium), which did not differ significantly from each other.

For the surface rugosity study, the design of the matrix considered a design that would facilitate the removal of the test specimen from the mold to avoid compromising the surface reading.

There was no statistically significant difference in the comparisons of surface contour differences between the master tooth-silver and the master tooth George Taub epoxy and the master tooth-polyurethane in one study conducted by Bloem *et al*.[28]

Authors determined the effect of liquids utilized as die lubricant on the compressive strength and surface hardness of a die stone. Silicone oil and palm oil did not affect both the compressive strength and surface hardness of die stone, whereas slurry water and water decreased both the properties of die stone.[3]

A significant difference in roughness was found by De Cesero *et al*.,[29] between Durone specimens at 1 hour and at 24 hours; however, the difference was not significant between 24 hours and 7 days.

The diametral compression tests were conducted 7 days after obtaining the samples (samples of dry plaster). Testing surface roughness, the type IV plaster Fuji Rock presented the best surface quality, whereas the plaster type IV Durone and Exacto-Form polyurethane resin showed no significant difference in surface roughness in relation to the matrix.

Polyvinyl siloxane as an impression material has been considered to be an excellent choice in terms of precision and dimensional stability.[30] Kumar *et al*.[31] and Valente *et al*.[32] agree that there is no significant change in the die with repeated pourings in the same mold. However, it shows some disadvantages as polymerization is inhibited by the use of latex gloves during its handling.[30,33] Thus, the polyvinyl siloxane used for the matrix impression was handled without latex gloves and the same impression was used for several dies.

Anusavice *et al*.[5] and Jayaprakash *et al*.[34] stress the importance of precision during the handling and dosage of die material with the use of distilled water on the mixture, which can interfere with the resistance and alter the die surface. In 2014, Tavarez *et al*.[15] showed that a 20% increase over distilled water recommended that volume does not interfere with compression properties of a die type IV. Azer *et al*.[35] could not find a difference between manual and mechanical spatulation under vacuum. Here, we used mechanical spatulation under vacuum.

According to Vohra and Habib,[20] handling of the polyurethane resin was challenging because the properties of the material are highly sensitive to the manipulation technique. The material is tacky and sticks to instruments, and has low thixotropy; therefore, it is necessary that pouring is carried out on maximum vibrations to avoid porosities.

Stone has limited transverse strength, which may predispose working casts to fracture when they are removed from impressions.[18]

Regarding resistance to fracture of plaster type IV, the setting time should be considered because it interferes with the mechanical behavior of tensile and compression strength.[1] Epoxy resin has four times the transverse strength of dental stone, so working casts of this dental material rarely fracture despite thinner areas.[18]

According to Anusavice *et al*.[5] recently poured models should be stored for at least 45 minutes. Here, we defined 60 minutes of rest before removing the test specimens from the impression, which is in accordance with Kim *et al*.[27] Chang *et al*.[7] showed small differences between different pouring time periods even for repoured casts. Sudhakar *et al*.,[10] also indicate that the hardness of die stone increased as a function of time.

Studies have shown that, the longer the storage time of the die material, the higher resistance to compression, approximately twice the value of that obtained after 1 hour (wet die samples).[5,17,29,36] The fast drying with microwaves achieves the properties found with drying at room temperature.[5,35] The diametral compression assays were carried out 7 days after the obtaining of the samples (dried die samples).

Both Kruskal–Wallis and Dunn's tests showed significantly higher diametral compression strength for Exakto-Form polyurethane resin (Bredent, Germany) in comparison to Durone type IV (Dentsply, Brazil) and Fuji Rock (Gc Europe, Belgium), which did not differ from each other in this aspect. De Cesero *et al*.[29] observed the mean compressive strengths of the various dental stone brands ranging from 26.67 MPa (Durone, 1 hour) to 65.14 MPa (Fuji Rock, 7 days).

Azer *et al*.[35] observed an increase in the diametric tensile strength (DTS) of Snap-Stone plaster (Type IV) from 1 hour to 24 hours.

The results from one study revealed that the storage time before repouring had less effect on the surface roughness than the materials themselves did.[7]

Surface rugosity tests showed higher surface quality for Fuji Rock type IV, whereas Duronetype IV and Exacto-Form polyurethane resin showed no significant difference from the matrix surface rugosity. Contrary to gypsum casts, which cannot reproduce details smaller than 20 µm due to its crystal structure,[25] polyurethane resin have the ability to reproduce details of up to 1–2 µm, and thus, Exakto-Form polyurethane resin showed the best performance to reproduce the fine details present in the model.

Conclusion

In this study, we show that the polyurethane resin shows the higher resistance to compression, and the Exakto-Form polyurethane resin showed better reproducibility of details than Fuji Rock type IV die.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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