Pesquisa Brasileira em Odontopediatria e Clínica Integrada 2019, 19(1):e4474
DOI: http://doi.org/10.4034/PBOCI.2019.191.87
ISSN 1519-0501

ORIGINAL ARTICLE

Injection Shrinkage and Water Sorption of Some Thermoplastic Dental Materials

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Academic Editors: Alessandro Leite Cavalcanti and Wilton Wilney Nascimento Padilha

Received: 24 October 2018 / Accepted: 17 April 2019 / Published: 06 May 2019

Abstract

Objective: To evaluate the change of diameter of different injection-molded thermoplastic materials. **Material and Methods:** Four thermoplastic injection-molded materials were analyzed (Bre.flex 2nd edition, Vertex ThermoSens, Perflex Biosens and Polyan IC). A total of 432 test samples were made in the form of an "hourglass". All samples were divided into three groups: Group I (Control) - consisting of 36 test samples for each type of material, that was not exposed to artificial aging or a wet environment; Group II - consisting of 36 test samples for each type of material, that were artificially aged through dehydration; Group III - specimen were subjected to artificial aging without dehydration. The diameter of each specimen was measured with a digital caliper. Data were analysed using the Student's t-test. **Results:** Regarding to shrinkage, the samples from the Bf Control group have a mean value of 1.56 mm and was observed a shrinkage of the injection-molded polyamide material within 0.25%. The comparison between the samples from Group II and Group III showed statistically significant differences ($p<0.001$). There were no significant differences between groups for Thermosens and Biosens ($p>0.05$). The comparison between Group II and Group III for Polyan IC samples shows that Group III has a higher arithmetic mean value ($p<0.01$). **Conclusion:** Shrinkage of the polymers during the injection process is present in all materials. The thermocycling and the storage in a dry or in a wet environment of the samples results in a change of the diameter in almost every single type of material.

Keywords: Dental Impression Materials; Dental Prosthesis; Denture Bases.

Introduction

The polymeric dental materials present in the oral cavity are exposed to a complex of factors: physical, chemical, and biological. These materials are used in an aggressive chemical environment such as saliva, and they are subjected to a high mechanical impact during the chewing process. On the other hand, the dental materials have a strong influence on the environment in the oral cavity and the body as a whole [1].

The contemporary methods of study give an opportunity to get a complete picture of the properties, structure, construction of the materials and their interaction with other materials and biological environments. The assessment of the properties of the materials has a great relevance with their application in dental practice.

The dimensional stability depends mainly on the polymerization shrinkage and water uptake. This one the most important properties and has a direct influence on the denture fitting. The shrinkage of the polymers is referred as the reduction of their volume during the cooling stage of their processing. This shrinkage is partly due to the difference in the densities of the melt and the cooled polymer in solid condition [2,3]. The shrinkage is a ratio and is expressed as a percentage.

The present study aims to evaluate the diameter change of test samples, made from four different injection-molded thermoplastic materials.

Material and Methods

As a result of 108 injection cycles, 432 test samples were made in the form of a "hourglass" with the help of a specially designed flask (Figure 1). The length of each sample is 35 mm. The test tube in the middle of the "hourglass" sample is 5 mm long and is 1.5 mm in diameter. The two cylinders in both ends of the test tube are 15 mm long and 5 mm in diameter (Figure 2). All test samples are made in accordance with the manufacturer specifications.

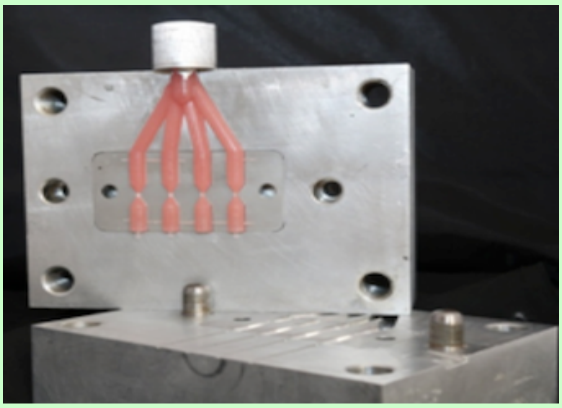

Figure 1. View of the specially designed flask.

Figure 2. View of the test samples.

The test samples were made from the following products: Bre.flex 2nd edition (Bredent GmbH & Co.KG, Senden, Germany), Vertex ThermoSens (Vertex Dental B.V., Zeist, The Netherlands), Perflex Biosens (Perflex LTD., Netanya, Israel) and Polyan IC (Bredent GmbH &

Co.KG, Senden, Germany). There were 108 samples for each material.The required number of samples was predetermined to obtain statistical reliability.

All 432 test samples were divided into three groups:

• Group I (Control): consisting of 36 test samples for each type of material, that is not exposed to artificial aging or a wet environment;

• Group II: consisting of 36 test samples for each type of material, that were artificially aged through dehydration;

• Group III: specimen were subjected to artificial aging without dehydration.

The following parameters was chosen for the thermocycling: temperature range 5-55^{0}C, submerging time of the samples in each bath - 30 seconds, intermediate drying time - 30 seconds and total of 5000 cycles. The total number of the thermocycles was divided into 20 phases with 250 cycles each and a single phase duration was 24 hours. In the period between the phases, the Group II samples were stored in a dry glass container at room temperature. Between the individual thermocycling phases the group III samples were stored in a glass container with distilled water at room temperature.

It was used the LTC 100 (LAM Technologies, Firenze, Italy) apparatus for the artificial aging of the test samples of thermoplastic materials (TMs). Before conducting the individual microtensile tests, the diameter of each specimen was measured with a digital caliper (Mannesmann Co., Düsseldorf, Germany).

The degree of shrinkage greatly depends on the type of polymer, but also on the conditions of the processing and shape of the item. According to the ISO 294-4 Standards, the shrinkage of the various polyamide materials in IP is within 0.5-1.5 [4]. The specially designed flask is made to compensate 0.3 - 1.0%, due to the fact that different groups of polymeric materials was used. The calculation of the shrinkage is made with the formula shr = /(D1/D)-1/:100, where D1 is the sample's diameter after the injection process, D is the sample's diameter set in the injection molding flask.

Statistical Analysis

Data were analyzed using IBM SPSS Statistics for Software, version 20 (IBM Corp., Armonk, NY, USA). Descriptive statistics were used to calculate the absolute and relative frequencies, mean standard deviation. Using the Student's t-test for normal distribution, the intra-group comparison was conducted.

Results

Bre.flex 2^{n}d edition (Bf) Samples

In Table 1 are shown the mean values of the test samples. The range of test samples in the Control group is the highest - from 1.50 to 1.60 +mm. The largest is the relative share of the specimens in Group II - up to 1.45 mm. In Group III, the measured results are located in the middle of the variation row. Measurement of the diameter of the test specimens in the Control group was done to determine whether a material shrinkage was observed during the injection process.

Table 1. Diameter of the test samples Bre.flex 2nd edition in the three groups.

Groups Size	Control Group		Thermocycling with Dehydration		Thermocycling without Dehydration	
	N	%	N	%	N	%
Up to 1.45 mm	-	-	17	47.2	-	-
1.46 – 1.47 mm	-	-	4	11.1	-	-
1.48 – 1.49 mm	-	-	4	11.1	4	11.1
1.50 – 1.51 mm	4	11.1	7	19.4	4	11.1
1.52 – 1.53 mm	8	22.2	-	-	8	22.2
1.54 – 1.55 mm	4	11.1	4	11.1	8	22.2
1.56 – 1.57 mm	12	33.3	-	-	12	33.3
1.58 – 1.59 mm	4	11.1	-	-	-	-
1.60 + mm	4	11.1	-	-	-	-
Total	36	100.0	36	100.0	36	100.00

The test samples from the Bf Control group have a mean value of 1.56 mm. Here it is observed a shrinkage of the injection-molded polyamide material within 0.25%, which is less than that in ISO standard. Table 2 shows the calculated arithmetic mean value is greater in the Control group, followed by Group II, and the lowest in Group III. For Group II, the mean value of the specimens' diameter is within 1.46 mm. The deviation from the Control group is clearly visible. The volume loss in this study group, after reading the difference in measured diameters, is within 6.43%. In Group III (thermocycling without dehydration of the test samples), the measured diameters are in the middle of the variation row, with a mean value of 1.53 mm. Compared to the Control group, the loss of volume is minimal, within 0.0 mm, or 1.9%. In compare to Group II there is an increase of the volume of the diameter by 0.7 mm, or 4.69%.

Table 2. Mean values of the diameters of the samples Bre.flex 2nd edition.

Groups	N	Mean (SD)	S_x	u	p-value
I (Control)	36	1.56 ± 0.49	2.93	16.97	<0.001[1]
II	36	1.46 ± 0.44	2.61	4.38	<0.001[2]
III	36	1.53 ± 0.51	3.04	10.43	<0.001[3]

[1]Comparison of the diameters between Control and Group II; [2]Comparison of the diameters between Control and Group III; [3]Comparison of the diameters between Group II and III.

Comparison of the Control group with Group II confirms the alternative hypothesis determined by the significantly higher mean value in the Control group (p<0.001). Statistically significant was verified in the difference in the mean values, where the highest value was registered in the Control group (p<0.001). The comparison between the samples from Group II and Group III showed statistically significant differences (p<0.001).

Thermosens (Ts) Samples

Figure 1 shows that the samples from three groups did not differ significantly in their diameter. The samples obtained from Ts of the Control group have a size with a mean value of 1.44 mm. The measured shrinkage of the material is less than 1%. In this observation was found a significantly higher percentage of shrinkage than the reported literature sources. The values of the measured diameters in Group II are almost identical to those of the Control group. This fact speaks of a lack of change as a result of the process of artificial aging. Only a certain difference was found in Group III, where the size of the specimens is in the largest range - from 1.42 mm to 1.49 mm.

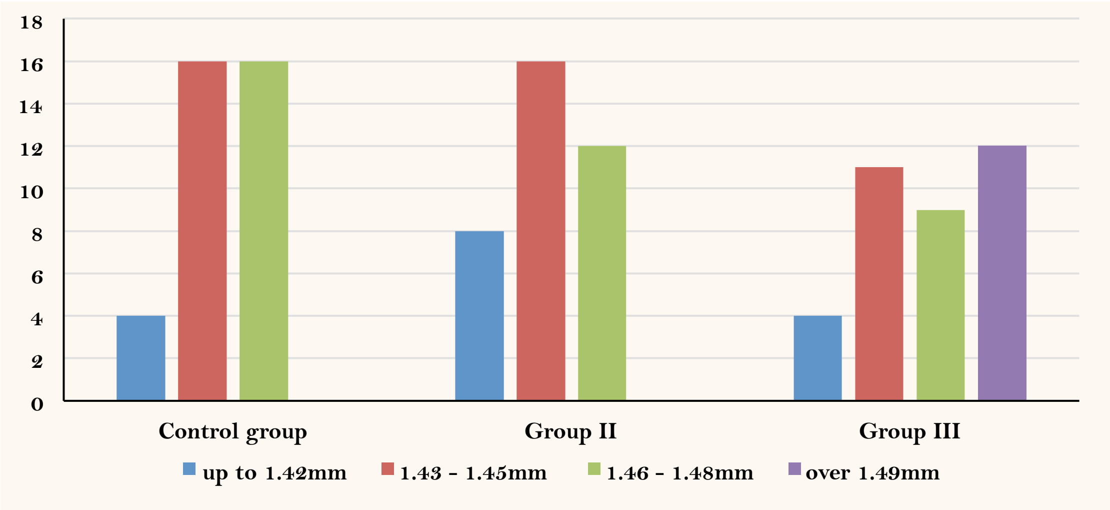

Figure 1. Diameter of the samples of Thermosens.

There were no significant differences between groups (Table 3).

Table 3. Mean values of the diameters of the samples of Thermosens.

Groups	N	Mean (SD)	S_x	u	p-value
I (Control)	36	1.44 ± 0.34	2.01	0.75	>0.05[1]
II	36	1.45 ± 0.69	2.22	0.55	>0.05[2]
III	36	1.47 ± 0.47	3.10	0.33	>0.05[3]

[1]Comparison between the Control group and Group II; [2]Comparison between the Control group and Group III; [3]Comparison between Group II and Group III.

Biosens (Bs) Samples

The distribution of the samples shows that the different study units are of different size, as presented in Figure 2. For the Control group they are in the range of 1.45 to 1.51 mm. The mean value is within 1.48mm. For this group there is a polymerization shrinkage above the standards. Group II is in the widest range - from 1.45 to 1.55 mm. In this group there was an increase in the diameter, within 1.5% compared to the Control group. Group III has the highest limits - 1.52 to 1.55+ mm. The increase in the measured value was 6.8% compared to the Control group and 6.7% and to Group II. There were no significant differences between groups (Table 4).

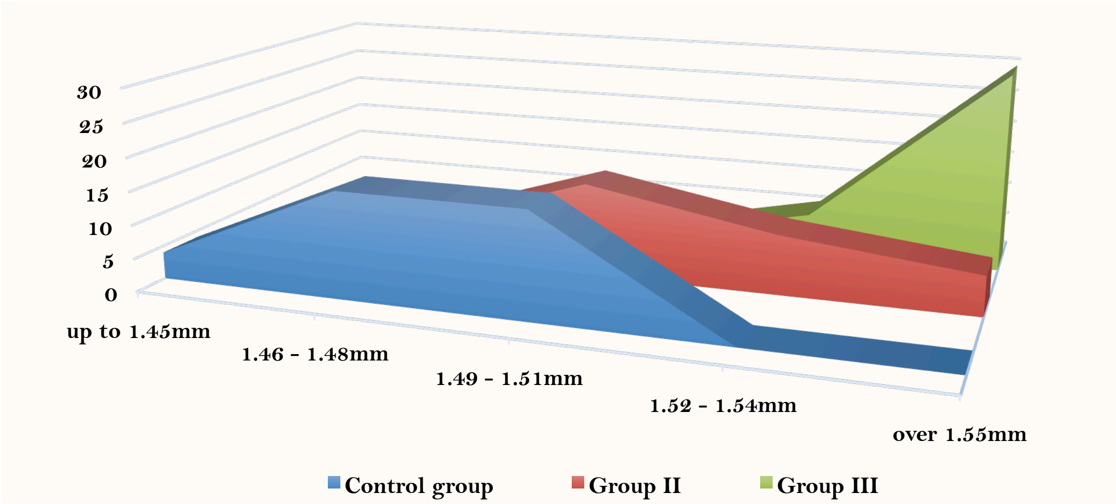

Figure 2. Diameter of the samples of Biosens.

Table 4. Mean values of the diameter of the samples of Biosens.

Groups	N	Mean (SD)	S_x	u	p-value
I (Control)	36	2.46 ± 1.11	2.22	0.02	>0.05[1]
II	36	2.56 ± 0.77	3.23	0.65	>0.05[2]
III	36	3.45 ± 1.54	2.24	0.55	>0.05[3]

[1]Comparison between the Control group and Group II; [2]Comparison between the Control group and Group III; [3]Comparison between Group II and Group III.

Polyan IC (Pl) Samples

The data concerning the diameter of the test specimens made by Pl are presented in Figure 3. In both Control group and Group II, the values for the test samples are in the range of 1.45 mm to 1.51 mm. The values from Group III - 1.48 mm to 1.57 mm are significantly higher. For the Control group, the mean value of the specimens' diameter is 1.46 mm.

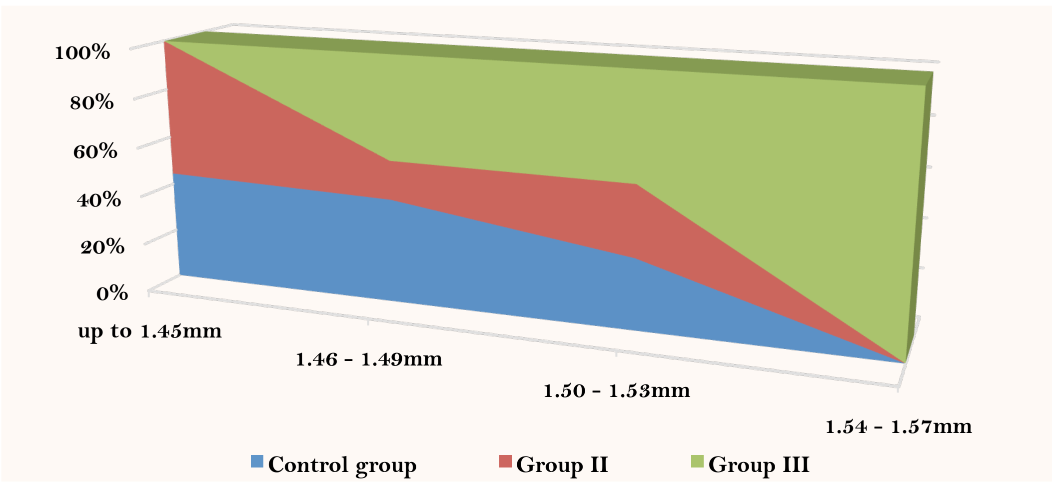

Figure 3. Diameter of the samples of Polyan IC.

The comparison of the arithmetic mean values calculated from the three comparison groups made by Pl from Table 5 shows that between the control and Group II (test samples subjected to thermocycling and dehydration), the mean values are completely the same. These results show that there is no change the arithmetic mean values of the measured diameters in Group II. The comparison of the Control group and Group III (test samples subjected to thermocycling and stored in distilled water) determines the alternative hypothesis, meaning statistically higher mean value for group III. The comparison between Group II and Group III shows that Group III has a higher arithmetic mean value (p<0.01).

Table 5. Mean values of the diameter of the samples of Polyan IC.

Groups	N	Mean (SD)	S_x	u	p-value
I (Control)	36	1.465 ± 0.02	0.012	0	>0.05[1]
II	36	1.465 ± 0.001	0.005	2.14	>0.05[2]
III	36	1.525 ± 0.02	0.013	3.00	>0.01[3]

[1]Comparison between the Control group and Group II; [2]Comparison between the Control group and Group III; [3]Comparison between Group II and Group III.

Discussion

For Bre.flex 2nd edition samples, the values from Table 1 shows that the shrinkage of the injection-molded polyamide material in the Control group is less than that in ISO standards [4] and would not have a significant impact on the accuracy of the product. The low shrinkage of the injected material results in a higher accuracy of the product and a lesser degree of deformation [5].

The volume loss in study Group II proves that the process of thermocycling and the storage of the samples between the phases in a dry container result in a reduction in the volume of the test specimens. The values for Group III show tendency of increasing the volume, when test samples are stored in a wet environment between the cycles.

Measurements of the samples diameter in Groups II and III give information about the presence of water absorption during the artificial aging process and during the different ways of storage between the individual thermocycling phases. The dentures made of thermoplastic materials spend a long time in a wet environment, which is the reason why water resistance and water absorption are very important properties. Moisture resistance refers to the ability of thermoplastic polymers to keep their properties in long-term water exposure. When in contact with the polymer, water diffuses through the surface inside the material, where the polymer swells and increases its volume. The absorption of water sometimes leads to change in shape of the product, decrease in strength and in other properties [6-8].

The process of thermocycling and storage in distilled water between the phases result in stability of the size of the sample diameter from Group III compared to the Control. When compared both groups subjected to artificial aging and different way of storage, there is a significant difference in diameters. This fact shows that the way of storage is essential for these materials.

Regarding the Thermosens samples, the increase in diameter of the test samples in Group over 1.49 mm means that the water absorption, which is within 1.5%, could be defined as minimal. Of a certain interest is the size comparison between groups II and III, shown in Table 3. There are no significant differences in the diameter from the mean values and unlike the other observed polyamide materials, the values are close.

For Biosens samples, the significant water absorption found in Group III confirms that way of storage is a factor for all polyamide materials. The comparison between the different groups, shown in Table 4, reaffirms the finding that artificial aging and storage have an impact on the properties of polyamide materials. This fact is supported by the previous results [9,10].

Regarding the Polyan IC samples, the results from Figure 3 shows that for the Control group the observed injection shrinkage is below 1% and a value of 0.2–0.8%, which is close to that defined by the ISO 294-4 Standard [4] and previous reports [11-13]. The storage in a dry environment of these materials has no influence on the polymer's volume. In Group III, the diameter of the specimens increased by an average of about 0.06 mm, suggesting that water absorption was clearly present in Group III and it is within 4.1%. The test sample's diameter for Group III was 4.1% bigger than that of Group II, which shows that the way of storage of this material has a significant effect on its properties.

Conclusion

Shrinkage of the polymers during the injection process is present in all materials. In the group of polyamides materials is the least, below the ISO Standards and in the group of Thermosens is the highest. In the acrylic polymer the shrinkage is within the standards. The thermocycling and the storage in a dry environment of the samples results in a change in the volume of the diameter, manifesting as a reduction in Bre.flex 2nd edition and an increase in Bre.flex 2nd edition and in Thermosens. The acrylic polymer material kept its volume.

The thermocycling and the storage in a wet environment greatly affect the size of the diameter for all materials observed. In the group of polyamide materials, the lowest water absorption was recorded at Bre.flex 2nd edition, and the highest at Thermosens. For Polyan IC, the survey data recorded a water absorption rate of up to 4%.

Authors' Contributions: BC, SY, RT, DI, and YK contributed to the study design, data analysis and interpretation, writing of the article, relevant critical analysis of the intellectual content and approval of the final version to be published

Financial Support: None.

Conflict of Interest: The authors declare no conflicts of interest.

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