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The influence of sterilization on the properties of the polyethylene used in biomedical applications

A. Ziębowicz [a,*], B. Ziębowicz [b], M. Dziekońska [b]

[a] Faculty of Biomedical Engineering, Department of Biomaterials and Medical Devices Engineering, Silesian University of Technology, ul. Gen. Charlesa de Gaulle'a 72, 41-800 Zabrze, Poland

[b] Faculty of Mechanical Engineering Materials, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

* Corresponding e-mail address: anna.ziebowicz@polsl.pl

ABSTRACT

Purpose: This paper presents results of studies on the effect of the sterilization process and aging process (for comparison) on the mechanical properties and the surface quality of low density polyethylene PE-LD used in biomedical applications.

Design/methodology/approach: In order to determine the changes in the surface structure of polyethylene PE-LD measurement of angle and roughness of samples were made. There were also measured mechanical properties - Shore hardness and tensile strength of PE-LD samples.

Findings: Results of this study indicate that the sterilization process and the aging process does not significantly affect the mechanical properties of polyethylene. These processes influence the structure of its surface, which is very important due to the its use in medical.

Practical implications: Low density polyethylene PE-LD is used in the manufacturing of laboratory equipment, such as syringes, gloves, laboratory dishes, catheters used in hemodialysis, connectors for the surgical drains, the surgical drains used in the treatment of sinuses, tracheostomy tubes.

Originality/value: Results are the base for further investigations of biomedical materials. Research are essential to search for new biomedical applications for polyethylene.

Keywords: Polyethylene PE-LD; Sterilization; Aging; Contact angle; Roughness; Mechanical properties; AFM

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PROPERTIES

1. Introduction

Polyethylene as one of the most used polymers is still expanding its application possibilities, also for biomedical applications [1-11]. The high density polyethylene PE-HD is used in medicine inter alia in the elements of hip, knee, shoulder joint endoprostheses, for non-biodegradable surgical threads [2, 3, 8]. Low density polyethylene PE-LD is used in the manufacture of laboratory equipment, such as syringes, gloves, laboratory dishes, catheters used in hemodialysis, connectors for the surgical drains, the surgical drains used in the treatment of sinuses, tracheostomy tubes [2, 3, 8].

Requirements for materials used by contemporary medicine is constantly evolving, differ from the requirements for materials in other fields of technical. In medicine, besides the appropriate mechanical and physico-chemical properties of the materials simultaneously is required biocompatibility, which among other things is realized by the sterilization-eliminating from their surface of harmful for human health microorganisms [12-17]. This process should lead to a certain level of sterility of the medical device. This is particularly important in the case of reusable medical devices used in the course of treatments and operations. Improperly made or completely abandoned sterilization is unacceptable, because it can lead to infection both medical staff and the patient, which in turn can lead to significant damage to health and even death. The sterilization process is standardized [18-19]. Very important is the selection of appropriate equipment, a sterilizing agent, humidity, pressure, and time of exposure of the product to the sterilizing agent. The sterilizing agent must be selected in such a way as to achieve its operation ensure the achievement of sterility in the planned conditions. Method of sterilization can be divided into physical and physico-chemical (Fig. 1).

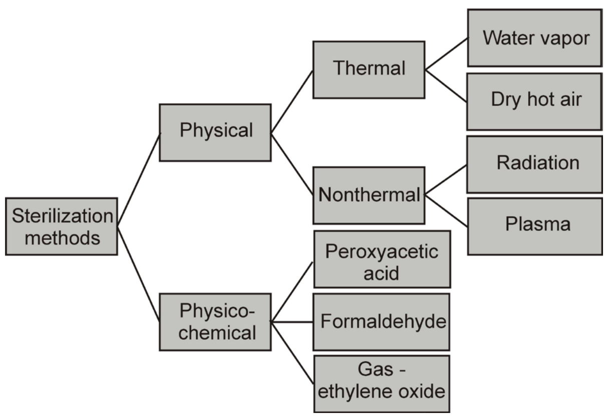


Fig. 1. General division of sterilization methods [12].

Among the methods for the sterilization can be distinguished one in which the temperature plays a significant role, as well as those where the main sterilizing agent is a chemical substance. Water vapor sterilization and dry hot air is a high-temperature sterilization, and the low-temperature sterilization is primarily plasma, radiation, or a gas. The properties of polymer materials can be changed under the influence of elevated temperature under the action of certain chemicals, and other factors (e.g. ultraviolet). Therefore, sterilization of polymer materials, including polyethylene, can be carried out only in particular methods, under appropriate conditions-usually low temperature methods because of their low thermal resistance. The most commonly used methods of sterilization of the polymers is ethylene oxide sterilization (this process is carried out at temperatures below 60°C) and sterilization using ionizing radiation [12-14].

The paper presents result of studies on the effect of the sterilization process on the properties of low density polyethylene PE-LD used in biomedical applications.

2. Material for research

The test samples were injection molded in an injection molding machine Krauss Maffei CX 50-180 using low density polyethylene PE-LD, which basic properties and applications are given in Table 1.

The tests were carried out on samples in the initial state (after the injection process), after sterilization process and for comparison after aging.

Sterilization of polyethylene was made in the Foundation for the Development of Cardiac Surgery in Zabrze. This process was carried out in an autoclave gas EOGas HW Series 4 Andersen company in temperature at 30°C. A gas cartridge was used, which contains ethylene oxide 17.6g. Exposure lasted 12 hours. The aging process was performed in a furnace Binder Company. The process parameters were as follows: temperature 70°C, aging time 24 hours. The temperature is selected so as not to exceed the softening point of polyethylene, which is 96ºC.

3. Research methods

In order to determine the changes in the surface structure of polyethylene measurement of angle and roughness of samples were examined. In order to determine its mechanical properties shore hardness and tensile strength of PE-LD samples were investigated. Five measurements were made in each case.

Table 1.
Characteristics of polyethylene used for testing

Low density polyethylene PE-LD Malen E FGNX, 23 D02		
Characteristic parameters	density	0.919-0.923 g/cm^3
	melt flow rate	2 g/10 min.
	softening temperature by Vicat	96^{o}C
	breaking stress	No less than 11 MPa
Method of obtaining	high pressure polymerization	
Characteristics of material	very high homogeneity, easy of processing, good optical properties, characteristic luster	
Methods for preparing	blow molding, extrusion	
Main areas of application	production of films with thicknesses from 25 to 50µm, injection processing, production of containers, possible use in contact with food, can be used in medicine	

Contact angle measurements were made by sessile drop method on the device Surtens Universal OEG Company. Volume of measuring liquid was 1µl, the measurement time was 60s. Roughness of polyethylene on the profilometer Surtronic 3+ Taylor-Hobson company by contact method was measured. Roughness R_a was calculated on the base of elementary stretch of 25 mm length. Hardness of the samples was examined by Shore method on hardness tester Sauter Company in a scale D. Tensile strength test was carried out on universal testing machine MTS Company Criterion 45. Tensile speed was 500 mm/min. In order to illustrate the topography of the surface atomic force microscope (AFM) XE-100 from Park Systems was used. In the study a non-contact mode of operation of the microscope was used. On the base of the images surface roughness R_a was also determined.

4. Results and discussion

Results of contact angle measurements are shown in Table 2 and Figure 2. Roughness measurements are given in Table 3. Results of hardness measurements in Table 4. and tensile test in Table 5 are shown. Figure 3 shows exemplary images of surface topography and the values of the surface roughness obtained by using the AFM.

Investigation results of contact angle show that both process sterilization and aging influence its reduction. In the initial state contact angle is 84.22°, after the sterilization process is 77.92° and 69.07° after aging (Table 2). The value of contact angle are associated with a surface roughness which is increased in relation to initial state from (0.47 µm) to value 0.63 µm after sterilization process and 0.70 µm after aging process (Table 3). The greater the roughness, the smaller the contact angle-better hydrophilic properties.

Sterilization processes and the aging does not influence the hardness of the polyethylene used for the examinations. Its hardness measured by Shore method in a scale D is approximately 45 (Table 4).

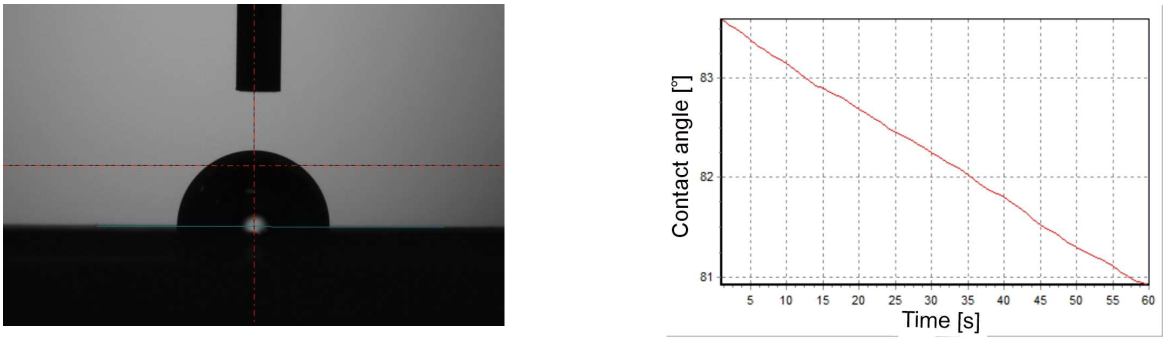
The results of the tensile test the polyethylene also indicates that the processes of sterilization and aging does not significantly affect the tensile strength which is approximately 12 MPa (Table 5). After test the samples elongation is reduced of about 20%. In the initial state is about 42.81%, after sterilization process 33.12% and after aging process 35.05% (Table 5).

The increase of wettability indicates the hydrophilicity of the polymer. This a factor favoring the colonization of most strains of bacteria on elements made of this material. Most bacteria living in the oral cavity has a high of the free energy, so they will be more likely to settle on hydrophilic surfaces [21-23].

Images obtained using AFM microscope (Fig. 3) also confirms a slight increase in surface roughness after sterilization processes and aging relative to the initial state. In the investigations of microareas the average roughness is 0.142 µm at initial state and 0.204 µm after the sterilization process, and 0.301 microns after aging. The differences in obtained results of roughness using the surface roughness profilometer and on the base of images from AFM microscope result from the size of the test area. In the case of AFM research, the area was 25 x 25 microns and was significantly lower than in profilometer research (section 25 mm).

Research of polyethylene mechanical properties after modifications associated with the sterilization process and aging does not indicate a change those properties. However, they influence on the quality of the polyethylene surface. Both after sterilization process and after aging process the roughness is increased and contact angle is decreased.

a)



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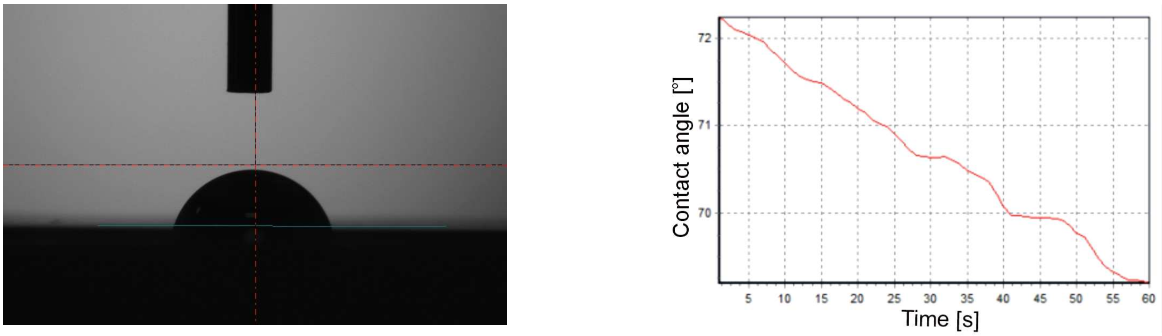

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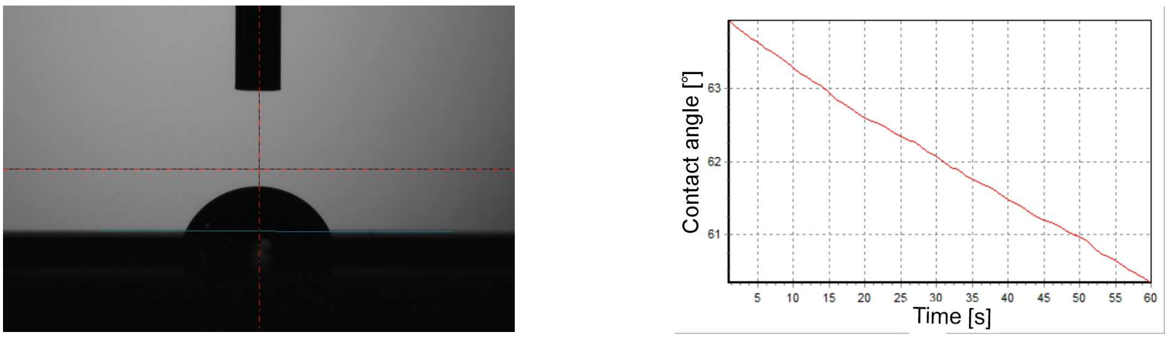

Fig. 2. Shape of the droplet on the sample and graph illustrating a change of contact angle versus time for the samples: a) in the initial state, b) after sterilization, c) after aging.

Table 2.
Results of contact angle measurements

State	Contact angle θ [º]					Mean value
	sample 1	sample 2	sample 3	sample 4	sample 5	
initial	84.49	74.70	93.07	88.13	80.69	84.22
after sterilization	75.66	73.66	74.35	83.21	82.73	77.92
after anging	72.32	69.67	68.53	71.15	63.69	69.07

Table 3.
Results of roughness measurements

State	Roughness R_a [μm]					Mean value
	sample 1	sample 2	sample 3	sample 4	sample 5	
initial	0.33	0.39	0.52	0.68	0.43	0.47
after sterilization	0.81	0.57	0.55	0.73	0.48	0.63
after aging	0.67	0.65	0.69	0.78	0.71	0,70

Table 4.
Results of hardness measurements

State	Shore hardness [°HSD]					Mean value
	sample 1	sample 2	sample 3	sample 4	sample 5	
initial	46.00	44.50	45.00	45.00	45.00	45.10
after sterilization	46.50	46.25	45.50	45.00	44.50	45.55
after aging	45.50	45.00	46.00	45.50	45.50	45.50

Table. 5.
Results of tensile test

State	Sample number	Tensile strength [MPa]	Mean value [MPa]	Young's modulus [MPa]	Mean value [MPa]	Elongation [%]	Mean value [%]
initial	1	12.50	12.04	118.47	114.52	35.40	42.81
	2	11.96		118.45		41.60	
	3	11.93		115.43		48.82	
	4	11.81		110.63		51.44	
	5	12.00		109.62		36.80	
after sterilization	1	12.17	12.17	111.34	116.37	42.54	33.12
	2	11.74		112.47		21.50	
	3	12.51		117.72		36.40	
	4	12.13		118.98		36.78	
	5	12.28		121.35		28.38	
after aging	1	12.61	12.49	121.22	114.59	32.64	35.05
	2	12.35		113.36		30.42	
	3	12.45		123.88		32.58	
	4	12.33		110.71		43.40	
	5	12.71		103.79		36.22	

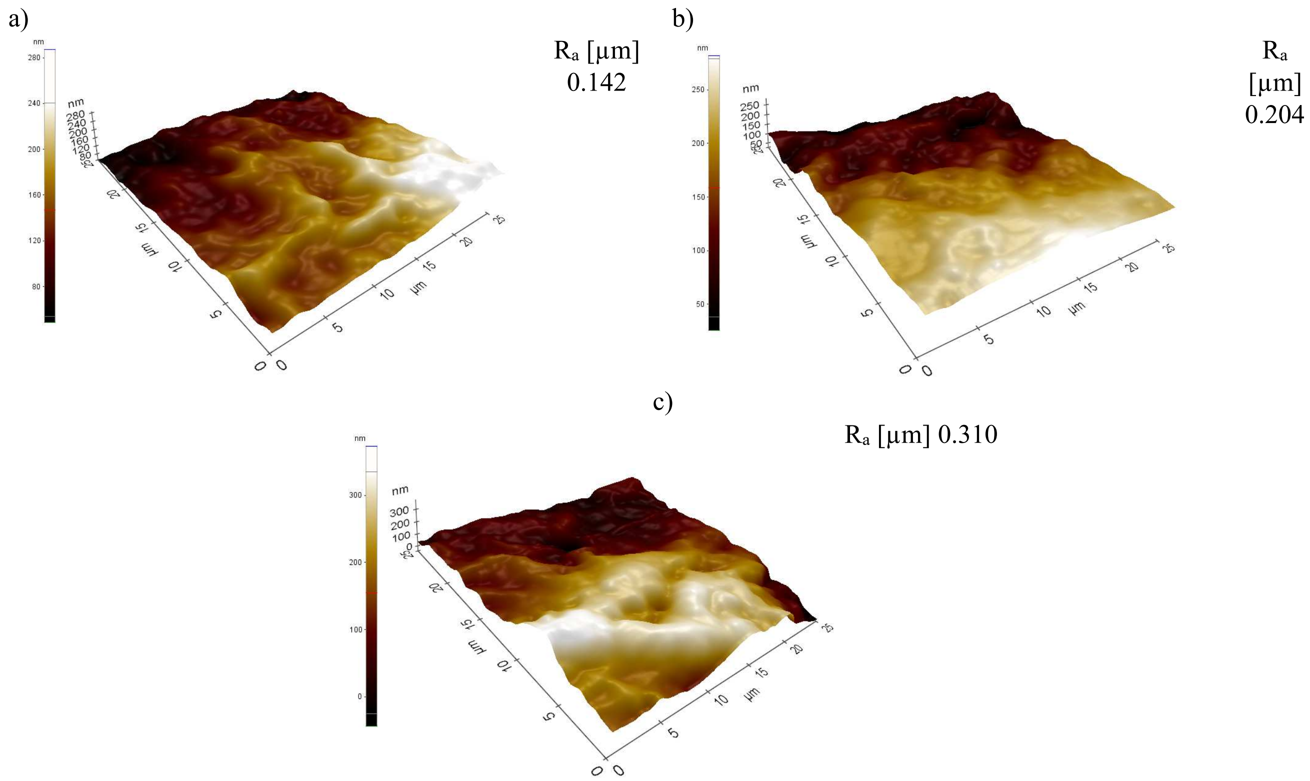

Fig. 3. Images of surface topography and surface roughness values obtained using AFM: a) in the initial state, b) after sterilization, c) after aging.

Polyethylene is a material widely used in medicine among other by the fact that it is cheap, easy in processing and in sterilization, is biocompatible in tissue environment, ensure the proper quality of the physicochemical and mechanical of products [2, 9, 20].

Polyethylene biomedical elements should always be subjected to a sterilization process not to cause any infection of patients. Due to the low thermal stability and chemical resistance of polyethylene it should, however, carefully choose conditions in which the its sterilization process is carried out to avoid possible changes in its structure, what can change its basic properties and hence the risk of its application [12, 21].

Research of mechanical properties of polyethylene after modifications associated with the process of sterilization and aging does not indicate a change those properties.

They influence on the quality of the polyethylene surface. Both after sterilization process and after aging process the roughness is increased and contact angle is decreased.

Used in research of sterilization with ethylene oxide is the most commonly used in hospitals to sterilize reusable medical equipment, and industrial scale for the sterilization of instruments and disposable medical equipment. Sterilization with ethylene oxide is subjected to the polymer elements of prosthesis, also applied to the stomatognathic system.

The increased wettability indicates the hydrophilicity of the polymer. It is a factor favoring the colonization of most strains of bacteria on elements made of this material. Most bacteria living in the oral cavity has a high of the free energy, so they will be more likely to settle on hydrophilic surfaces [21-23].

Sterilization of polymer materials can't affect the material by changing their chemical structure and by modifying their surface topography. Material subjected to a sterilization process should have a controlled roughness, contact angle and respectively to apply surface preparation.

The useful life of polymer materials depends largely on the temperature which they are subjected to. Both sterilization and aging proposed in the paper are thermally-activated processes and significantly affect the range of possible applications of these materials.

The main goal of sterilization is to achieve a certain level of sterility. At the same time it can not affect the structure of the material surface and its interior. If this situation took place, other non-temperature (less popular) sterilization method should be used, which in less degree will affect the structure of the material and the maximum way will reduce unwanted microbes from the surface.

5. Conclusions

Used in research, the sterilization with ethylene oxide, is the most commonly method used in hospitals to sterilize reusable medical equipment, and in industrial scale for the sterilization of instruments and disposable medical equipment. Sterilization by ethylene oxide is subjected to the polymer elements of prosthesis, also applied to the stomatognathic system.

Sterilization of polymer materials can't affect the material by changing their chemical structure and by modifying their surface topography. Material subjected to a sterilization process should have a controlled roughness, contact angle and proper to apply surface preparation.

Based on the study the following conclusions can be shown:

- After aging and sterilization proesses the tested polyethylene shows higher surface roughness compared with initial state.
- Application of the aging and sterilization processes increases the wettability (hydrophilic property) of polyethylene.
- The sterilization and the aging processeses do not substantially affect the tensile strength (reduced only by 20% elongation) and the hardness of polyethylene.

References

[1] W. Szlezyngier, Plastics-chemistry, manufacturing technology, properties, processing, application, Volume 1, Fosza Educational Publishing House, Rzeszow, 1998.

[2] J. Rabek, Contemporary knowledge of polymers, Scientific Publishers PWN, Warsaw, 2008.

[3] I. Gruin, Polymer materials, Scientific Publishers PWN, Warsaw 2003.

[4] J. Pielichowski, A. Puszyński, Polymer Chemistry, Science Publishing-Technical Thesis, Kraków, 2004.

[5] W. Loose, Introduction to Polymer Science, AGH College Educational Scientific Publishing, Kraków, 1999.

[6] S. Penczek, Z. Florjańczyk, Polymer Chemistry, Volume III, Publishing House of the Technical University of Warsaw, Warsaw, 1998.

[7] C.I Chang, Extrusion of Polymers: Theory and Practice, Hanser Publications, Munich, 2000.

[8] E. Olędzka, M. Sobczak, W.L Kołodziejski, Polymers in medicine-a review of past achievements, Polymers 11-12 (2007) 795-803.

[9] J. Marciniak, Biomaterials, Silesian University of Technology, Gliwice, 2002.

[10] J. Marciniak, M. Kaczmarek, A. Ziębowicz, Biomaterials in dentistry, Silesian University of Technology, Gliwice, 2008.

[11] S. Błażewicz, L. Stoch, Biomaterials, Volume 4, Exit, Warsaw, 2003.

[12] P.R Murray, K.S Rosenthal, M.A Pfaller, Medical microbiology, Mosby Elsevier, Philadelphia, 2009.

[13] Z. Huys, M. Kopyra, Sterilization of medical resources, Polish Association for the Development of Medical Sterilization and Disinfectant, 2009.

[14] Sterilization of health care products-Biological indicators-Part 2: Biological indicators for ethylene oxide sterilization processes BS EN ISO 11138-2, Polish Committee for Standardization, 2001.

[15] A. Jaguś, Low-temperature sterilization with ethylene oxide in questions and answers, Nurse Epidemiology 1/52 (2013) 85-89.

[16] W. Głuszewski, Z. Zagorski, Radiation sterilization of medical devices, Contemporary Oncology 7/10 (2003) 787-790.

[17] M. Podrez-Radziszewska, I. Głuszewski, Radiation modification of polyethylene surgical implants, Contemporary Oncology 9/8 (2005) 365-367.

[18] BS EN ISO 17664: 2004, Sterilization of medical devices-Information supplied by the manufacturer for re-sterilization process for medical devices.

[19] PN-EN ISO 14937: 2004, Sterilization of health care products-General requirements for characterization of a sterilizing agent and the development, validation and routine control of a sterilization process for medical devices.

[20] R.G. Craig, Dental Materials, Elsevier Urban & Partner, Wroclaw, 2008.

[21] R. Pokrowiecki, S. Tychy, M. Zaleska, Perigraft infection Problems, Progress microbiology 2/53 (2014) 123-134.

[22] E. Sobolewska, B. Frączak, S. Błażewicz, K. Senko, M. Lipski, The wetting angle comparison of the basic materials used in fabrication of prosthetic dentures in vitro, Prosthodontics 6 (2009) 401-406.

[23] M. Dubiel, S. Łagan, Evaluation of contact angle and surface free energy of the different types of dental materials, Current problems of Biomechanics 7 (2013) 39-42.