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Accelerated Ageing of Implantable, Ultra-Light, Knitted Medical Devices Modified by Low-Temperature Plasma Treatment - Part 2. Effect on Chemical Purity

Abstract
The impact of simulated storage conditions (accelerated ageing) for the chemical purity of innovative ultra-light textile implants (knitted) designed for use in urogynaecology and general surgery (procedures in the treatment of female incontinence, in hernia treatment and vagina plastic surgery) was estimated. The chemical purity of the knitted implants designed: untreated and with low-temperature plasma surface treatment in the presence of the fluoroorganic compounds was estimated. The acceptability of the risk related to the impact of storage conditions on the chemical purity of the implant products was simulated. The examination was based on Standard PN-EN ISO 10993-18:2008: "Biological evaluation of medical devices - Part 18: Chemical characterisation of materials" and was assessed in accordance with Polish and European standards.

Key words: *ultra-light knitted implants, accelerated ageing, chemical purity, leachable substances.*

Introduction

Impressive progress can be seen in modern implant surgery. Yet more discriminating quality requirements are set before materials to be used in implants, fostered by the increasing demand. The lowering age of implant users is another factor calling for high quality, which results in novelty materials being sought, made according to the most advanced technologies to provide as high as possible connection between the implant and substituted part of the body and its function. This is why modern medical devices are the most expensive man-made materials [1, 2].

There is a wide range of medical applications for resins thanks to their different properties when compared to metallic and ceramic materials. Good forming ability, easy sterilisation, bio-inertness, non-allergenic and non-toxic action, and adequate physical-chemical properties are amongst the beneficial qualities and behaviour of polymers [3].

Polypropylene (PP) is an implantable material which has recently found wide use mainly in non-resorbable surgery meshes for the reconstruction of soft tissue defects in hernia repair. The use of polypropylene meshes was a real breakthrough in the treatment of connective tissue defects in the abdominal and inguinal surgery. It enables a tissue tension-free operation, thus largely reducing pain and shortening hospitalisation.

Knitted fabrics find ever wider application in medicine by improving patients' health, thanks to features like high tenacity, low density, lack of water imbibition and good healing properties.

Research made so far in the designing of the medical devices from innovative, polypropylene, ultra-light knitted fabrics for use in urogynaecology (procedures in the treatment of female incontinence and vagina plastic surgery) and general surgery (hernia treatment) has met the expectations of medicine and patients themselves [4 - 12].

Non-resorbable meshes made of polypropylene multi -or monofilaments (class IIB) are designed for hernia surgery. They have been available world-wide for many years, for example in Poland under the trade name, OPTOMESH®PP, DALLOP.

Urology polypropylene bands have long since been used in the surgery for female incontinence (market product DALLOP®NM, class IIB).

One of the main disadvantages of the implants is a high surface density reflected in the high mass of the synthetic material implanted, which may be the reason for a local chronic reaction. Therefore lighter implants with a sufficient tenacity are strived for. Another aspect of textile implant structures is the one-side developed surface, which promotes the growing through of connective tissue and provides better fastening of the implant, particularly in suture-less surgery. Moreover in the scope of the investigation, low–temperature plasma treatment of the surface of knitted implants was applied, which was expected to reduce the risk of complications related to the adhesion of the implant to internal organs, particularly in low-invasive surgery. The main quality demands that are set before the implant materials are as follows: a fast growing-through of the tissue, quick healing, biocompatibility, and lack of irritation of human tissue.

The reaction of the human organism to a foreign body is distinctly reduced whenever ultra-light, non-resorbable polypropylene monofilament meshes (trade name OPTOMESH™, Optilene® Mesh LP) are used in hernia repair. The monofilament structure minimises the risk of bacterial infection.

The present investigation is aimed at a possible reduction of the irritating action of the polymeric material introduced to the organism by applying ultra-light polypropylene monofilament knitwear of possibly low surface density and very fine spatial structure, which has the effect of introducing a minimised amount of synthetic material to the patient's organism.

Chemical purity and its stability in medical devices is a key issue concerning biocompatibility. It is assumed that materials

which under simulated conditions deliver chemical substances may, though not necessarily, cause a local reaction of the tissue into which they are implanted. Lack of the implant's biocompatibility causes a complex, difficult- to-define reactions which, in the extreme, may lead to the rejection of the implant. This, together with the synergy and complexity of the phenomena proceeding in the course of the migration of substances from the implant, is an obstacle in defining parameters that would quality- and quantity-wise limit the chemical substances. Characteristics of the chemical substance leached from the implant is solely a support in defining the quality base for modified and commercial medical devices, including a stability assessment of the quality and quantity level (assessment in accelerated ageing examination and real ageing). Knowledge of the chemical composition and, what is more important, of the qualitative and quantitative composition of the substance which migrates in the course of simulated clinical use does not permit, particularly in the case of newly developed or much modified medical devices, to relate to biocompatibility. The latter is to be tested in the course of in vivo and in vitro examination as defined in ISO 10993-1.

Ultra-light textile implants that come into contact with tissue and body fluids must satisfy specific demands set for biomaterials like material biocompatibility. The last is manifested by the fact that the material is chemically and immunologically neutral and does not reveal any toxic or destructive action in the given environment of the organism.

When designing implant materials, complying with demands like mechanical-, useful- and physical-chemical properties is a precondition but does not guarantee success. Really crucial is the response of the cells that approach the implant's surface.

Ultra-light textile implants are 3D medical structures with a three-direction orientation of the polypropylene fibers (medical-grade monofilament - class VI according to American Pharmacopoeia). Medical grade PP features the lowest density amongst commercial polymers; it is classified as a neutral polymer, meaning that it contains only a minimal amount of auxiliary substances which are neither delivered to nor degrade in a biological environment.

PP is an inert polymer without covalent bonds in the chain, which is why the surface of PP implants requires functionalisation. It was in the prototypes of the medical devices designed that this was accomplished by low-temperature plasma surface modification in the presence of a low-molecular fluoroorganic compound (the fluorocarbon polymer layer deposited). That kind of modification, by the depositing of a thin layer (25 - 50 Å), affects the quality of cell response, biocompatibility and hydrophilic properties; moreover it accelerates the adhesion process and proliferation [13 - 15]. The active surface layer of the implant not only enables the tailoring of surface properties such as wettability and surface energy but also provides the chance of controlling the material degradation process [16].

A very important aspect is the adapting of chemical properties including the quantitative and qualitative profile of leachable substances, and the physical properties of the implant material surface. This results in biocompatibility and the possibility to stimulate tissue regeneration, which increases the chances of the better acceptance of the implant in vivo [17].

One unavoidable part of the research is the assessment of the clinical risk of using polymeric materials as implants by analysing the mechanisms of degradation and assessing quantity-wise the degradation products that may be delivered in the course of chemical reactions, migration and depolymerisation.

It was also important in the investigation to learn the influence of storage conditions of the final product designed upon the quantitative and qualitative profile of leachable substances determined in the testing of chemical purity. Therefore the method of accelerated ageing is being used more and more for designed medical devices to anticipate risk defined as a profile of potential leachable substances in conditions of simulated storage and clinical handling [18].

In this work, as a part of the chemical purity assessment of the implantable medical devices designed, the profile of leachable substances under simulated conditions of use (specific processing conditions, storage, nature and contact time of the product) was estimated on the basis of EN and ISO standards harmonised with European Directives concerning medical devices [19 - 23].

The aims of the work were as follows:
- determination of the impact of simulated storage conditions on the chemical purity of the prototype knitted implants designed: (a) surface-modified by low-temperature plasma in the presence of a low-molecular fluoroorganic compound and (b) unmodified ones;
- the assessment of risk acceptability concerning the impact of storage conditions on the chemical purity of the medical devices designed.

Selected prototypes of the medical devices designed were subjected to accelerated ageing according to a research programme based on guidelines of Standard ASTM 1980F:2002. The programme was an extension of research published earlier [6 - 9] concerning biomechanical and chemical properties.

Materials

Raw-materials

Knitted medical devices for hernioplasty and vaginoplasty were designed using polypropylene monofilament fibres with a diameter of 0.08 mm (linear density of 46 dtex) with properties as described in [21].

Design of implantable medical devices

Knitted medical devices for hernioplasty and vaginoplasty were designed and optimised at the Knitting Department of the Lodz University of Technology based on the outcomes of earlier experiments [17, 21].

Low- temperature plasma surface modification of implantable medical devices designed

One-side modification of the knitted implants prepared was made at the Department of Commodity, Material Sciences and Textile Metrology of the Lodz University of Technology by low-temperature plasma treatment with fluorine organic derivatives (tetradecafluorohexane/Fluka) using the CD 400PLC ROLL CASSETTE Plasma system, Europlasma, Belgium. Details of the procedure of the modification process are described in [18] according to Standard [23].

Finishing of the implantable medical devices

The finishing process including the steam sterilisation of the knitted implants modified by low-temperature plasma with tetradecafluorohexane (code PF) is described in [18] according to Standard [23]. The knitted implants, identical in structure but untreated by the low tem-

perature plasma (code KO), were finished the same way.

Prototypes of the knitted implants (both unmodified and modified by low temperature plasma treatment) were packed in a double medical grade packaging system adaptable for steam sterilisation (OPM/ Poland) as described in [18] according to Standard [23].

Altogether 130 pieces of KO and PF implants were prepared as a prototype batch for testing of accelerated ageing. Samples for testing were taken statistically from the whole knitwear batch, prepared on a semi-industrial scale and resembling typical industrial production.

Methods

Accelerated ageing

The testing of accelerated ageing was designed on the basis of Standard ASTM F1980:2002: Standard Guide for Accelerated Ageing of Sterile Barrier Systems for Medical Devices. This document specifies guidelines for the accelerated ageing testing of medical packaging. However, it can be easily adapted to the accelerated ageing of medical devices (considering similar potential hazards) to assess the influence of storage conditions upon functional properties and the safety of newly designed medical devices. The medical devices designed placed in typical packaging (direct packaging satisfying quality requirements of Standards PN-EN ISO 11607-1:2011 and PN-EN 868-5:2009) were tested after steam sterilisation in validated industrial conditions. The accelerated ageing was performed in a KBF 240 chamber (Binder GmbH/ Germany), where the elevated temperature was the factor simulating accelerated ageing. The temperature of the chamber was 60 ± 2 ºC and the RH 20 ± 5%, under which conditions the medical devices were kept for 28 days (simulation of 1 year of ageing) and 56 days (simulation of 2 year's ageing).

The residence time of the medical devices in the chamber was calculated using the Arrhenius equation (ASTM F 1980:2002, p. 7.3.), adopting the value of 3.7 as the ageing factor. The testing was performed at the accredited Laboratory of Metrology of the Institute of Security Technologies "MORATEX", Lodz, Poland. Both plasma-modified (code PF) and unmodified (code KO) implants were tested.

Estimation of chemical purity

Parameters of the chemical purity were estimated based on results obtained in the risk analysis and on directives of Standards PN-EN ISO 10093-18:2009 and PN-EN ISO 10993-1:2010.

Exhaustive extraction and three-step extraction

The content of leachable substances (profile of leachable substances) was estimated by the methods of exhaustive and three-step extraction. The first was accomplished on Soxhlet apparatus with petroleum ether according to the procedure specified in Standard PN/P-0607:1983.

The three-step extraction was made in accordance with directives given in Standard EN ISO 10993-12:2009, where the following solvents were used in turn:

- purified water (water for injection by Baxter Co);
- 2-propanol according to the method given in Standard PN/P-04781/06;
- petroleum ether.

Extraction module: 10 g of test material/100 cm^3 of the extraction substance, based on guidelines contained in Standard PN-EN ISO 10993-12:2007 and in accordance with the 2009 revised edition of the Standard PN-EN ISO 10993-12 (Table 1) . Standardised surface area and volume of extraction fluid for low-density materials.

Preparation of aqueous extracts for estimation of chemical purity (analysis of the profile of leachable substances)

The aqueous extract was prepared with the following module: 10g of fine cut pieces of the material about 1 cm long on 100 cm^3 of water for injection (Baxter Co) as per directives of Standard PN-EN ISO 10993-12:2009.

Steam sterilisation was made at 121°C for 60 minutes (p.10.3.1 of the standard; critical conditions for designed implants).

pH estimation in the aqueous extracts

The pH reaction of the aqueous extracts was measured in accordance with Standard PN-EN ISO 3071:2007 by a LAB 860 SET pH-meter (Scott, Germany) equipped with a BluLine 14 pH electrode with an integrated temperature sensor for measurements in pure fluids with an insignificant amount of the deposit.

Estimation of the turbidity of the aqueous extracts

A method for turbidity measurements in aqueous extracts was prepared on the basis of a visual method described in Polish Pharmacopoeia, the VII edition. A suspension of formazin was used as a basic turbidity reference equal to 4000 NTU (*Nephelometric Turbidity Units*), which is a blend of hydrazine sulfate and hexa(methylenetetramine) (urotropin). The visual method was applied in instrumental turbiditymetric measurements with the use of a spectrophotometer - Unicam 5625 UV/VIS, USA. A calibration curve of the basic turbidity pattern was determined from five comparative suspensions: I, II, III, IV & V, representing the turbidity degrees of 3, 6, 18, 30 and 45 NTU. An analytical wave length of λ = 400 nm of the light was adopted. The turbidity degree of the comparative suspensions was estimated by measuring the scattered light compared to purified water as reference. A rectilinear plot was able to be drawn of the turbidity degree determined for the comparative suspensions. A maximal turbidance of 0.171 was seen for comparative suspension No. V.

The aqueous extract is considered translucent if its translucence matches that of water measured at the same conditions or if its opalescence does not exceed that of comparative suspension No II, equal to 6 NTU.

Determination of heavy metal ions in the aqueous extracts

The content of heavy elements: cadmium, chromium (sum of all oxidation states), lead, zinc, and mercury in the aqueous extracts was determined by Atomic Absorption Spectroscopy using a SCAN-1 spectrometer made by Thermo Jawell ASH. Cadmium, chromium, lead and zinc were determined directly in the aqueous extracts by the flame method ASA (FAAS*) at the following parameters:

- Cd: wave length λ = 228.8 nm, flame acetylene-air, limit of determination – 0.02 mg/dm^3
- Cr: wave length λ = 357.9 nm, flame acetylene-N_2O, limit of determination – 0.2 mg/dm^3

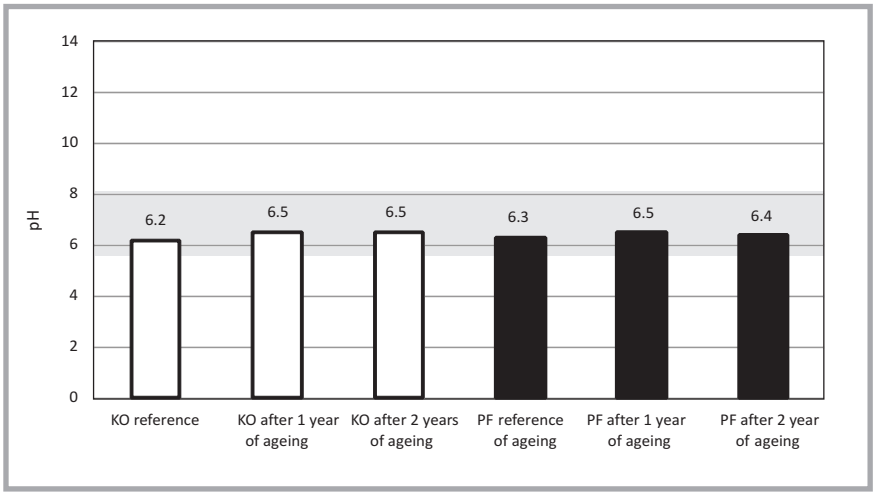

Figure 1. *Values of pH of aqueous extracts prepared from reference samples and from prototype knitted implants KO and PF subjected to ageing simulated by acceleration. Limits of the pH permissible in respect of operational safety are marked in the graph.*

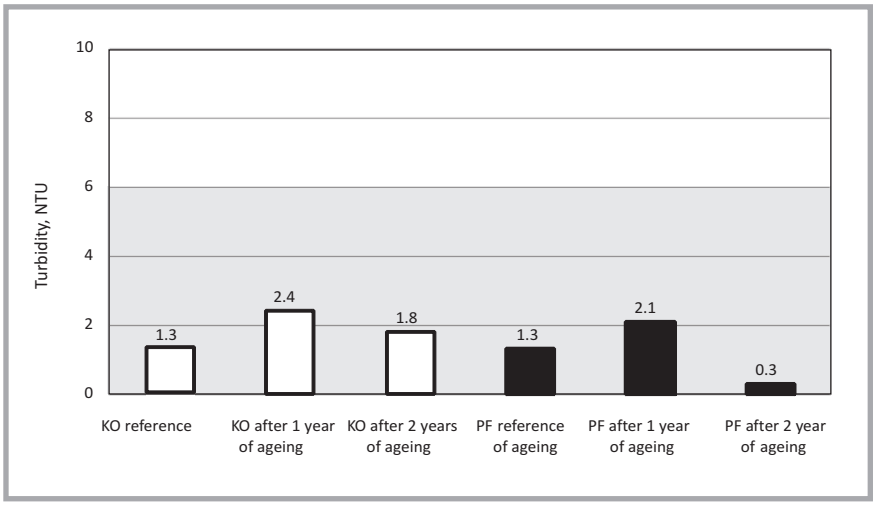

Figure 2. *Change in turbidity degree in aqueous extracts prepared from reference samples and prototype textile implants KO and PF subjected to simulated accelerated ageing.*

- Pb: wave length λ = 217.0 nm, flame acetylene-air, limit of determination – 0.2 mg/dm^3
- Zn: wave length λ = 213.9 nm, flame acetylene-air, limit of determination – 0.01 mg/dm^3.

Mercury was determined by the method of cold vapour generation ASA (CVAAS) using a device for the generation of cold vapours - Atomic Vapor Accessory 440, made by Thermo Jawell ASH, at the following parameters: wave length λ = 253.7 nm, reductive solution 5% $SnCl_2$ in 20% HCl, carrier gas – Ar, and limit of determination – 0.01 mg/dm^3.

Determination of the permanganate value (oxidability) of the aqueous extracts

The permanganate value was determined according to the directives of Standard PN-P-04896:1984. Water for injection (Baxter Co) served as reference: it was subjected to the same processing conditions as the sample tested.

Determination of chloride ion content in the aqueous extracts

The content of chloride ions in the aqueous extracts was determined by the visual method described in Standard PN-P-04895:1984.

The method employs argentometry titration of the aqueous extracts prepared with a 0.01 mol/dm^3 $AgNO_3$ solution in the presence of chromium ions. The determination limit of the method is 0.003 mg of $[Cl]^-$ ions /1g of the material tested. A 3.0% hydrogen peroxide solution was added at the end of titration to enhance the hue intensity at the colour change point.

$[Cl]^-$ free injection water (Baxter Co.) was used to prepare the reference and solutions needed for the determination of chloride ions i.e. solutions of potassium chromate, hydrogen peroxide and a standard volume solution of silver nitrate.

Results and discussion

For the textile implants designed for use in urogynaecology and general surgery, results are presented of the examination of selected physical-chemical quality parameters which directly affect the operational safety, especially when it comes to biocompatibility. Results of the parameter testing are compared with reference standards (KO and PF) of prototypes taken directly from the manufacturing process, which permitted to estimate the changes in chemical purity of the products proceeding in the course of ageing.

pH of the aqueous extracts

Figure 1 presents pH values of aqueous extracts prepared from KO and PF implants after 1 and 2 years of accelerated ageing compared with the reference.

The pH values estimated for the starting prototype implants designed for hernia and vagina repair, both modified with low-temperature plasma and unmodified, amount to 6.2 (KO) and 6.3 (PF). pH values of aqueous extracts of the prototype medical devices, both modified and unmodified, after 1 and 2 years of accelerated ageing do not differ much from the starting value (reference); the value of pH is close to a neutral reaction and to that of human skin, actually falling into the optimal range for that type of medical device.

The results confirm the absence of substances that could affect the change in pH reaction by migration after long storage of the medical devices. It must be stressed that a pH below 4 and above 8 brings about the risk of irritation of the surrounding tissue, which in extreme cases could be the reason for implant rejection or cause the forming of a thick cartilage capsule, leading to a stiffening of the implant locality

Turbidity of the aqueous extracts

Figure 2 presents changes in the turbidity of aqueous extracts prepared from the test implants proceeding in the course of accelerated ageing.

The permissible safety level of the turbidity of the aqueous extracts is marked in the graph (as laid down in Polish PharmacopoeiaVII).

The turbidity of aqueous extracts prepared from the knitted implants did not remarkably change during accelerated ageing in comparison with the starting material, and its value did not exceed the limit of 3.

According to Polish Pharmacopoeia VII, recommendation for medical devices, a solution is considered transparent if the turbidity does not exceed the value of 6 NTU. In view of the results presented above, it may be concluded that in the course of accelerated ageing no changes occur in the PP polymer which would promote the delivery of dulling substances to an extent hazardous to operational safety. The low-temperature plasma treatment slightly reduced the turbidity of the aqueous extract as result of the pretreatment of the fibre surface prior to the modification [18, 23].

Content of $[Cl]^-$ ions in the aqueous extracts

Chloride ions are one of the most serious impurities imparted to textile medical devices during manufacture, for example by the addition of auxiliary agents in the textile processing or remaining after the polymer synthesis. The substances tend to degrade in the product. Monitoring of the amount of $[Cl]^-$ ions migrating during storage is crucial for the safety of the medical devices and also permits to assess the potential risk of the delivery of chloride derivatives from the implanted device.

The change in chloride ion content in aqueous extracts of the starting material and implants designed, KO and PF, after 1 and 2 years of accelerated ageing is presented in ***Figure 3***.

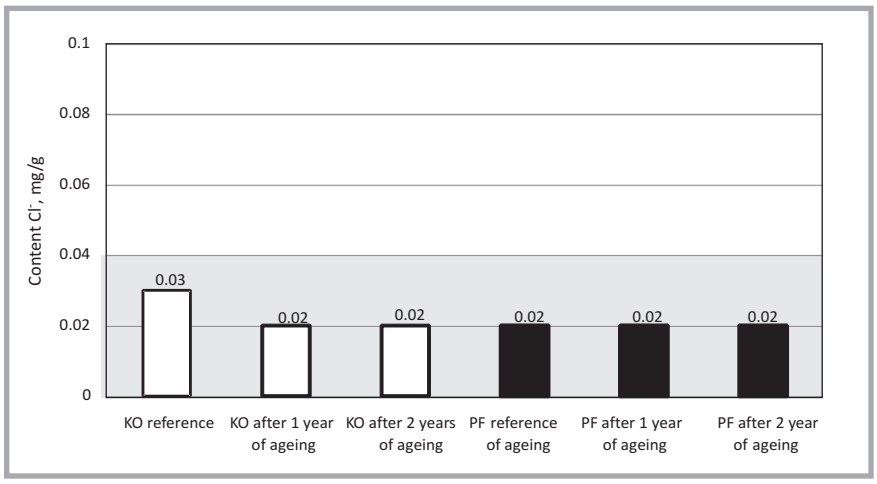

Figure 3. *Change in chloride ion content in aqueous extracts from the reference samples and prototype implants KO and PF subjected to simulated accelerated ageing. The permissible safety level of chloride ion content in the aqueous extracts is marked in the graph.*

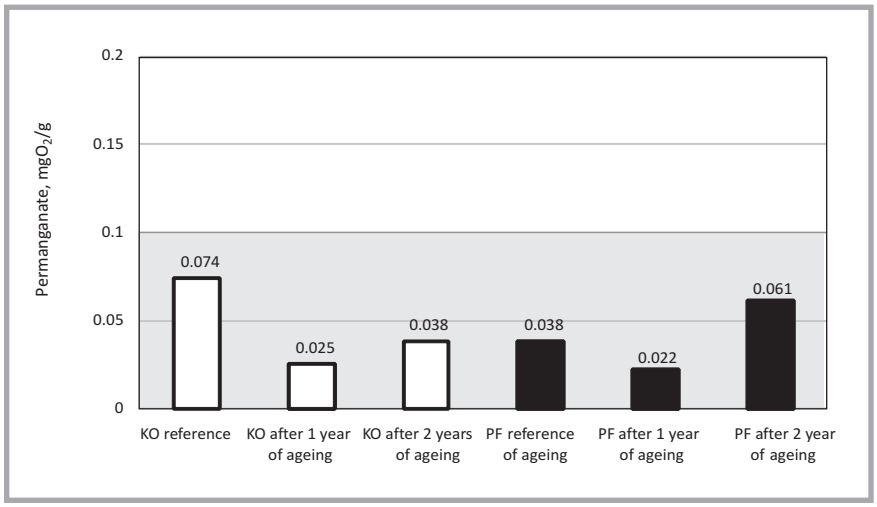

Figure 4. *Change in permanganate value of aqueous extracts from reference samples and prototype implants KO and PF subjected to accelerated ageing.*

Table 1. *Heavy metal content in aqueous extracts from the reference samples and prototype implants KO and PF before and after accelerated ageing.*

Content of heavy metals		KO			PF		
		Reference	Time of ageing after,		Reference	Time of ageing after,	
			1 year	2 years		1 year	2 years
Cd	mg/100 cm³ of the extract	< 0.02			< 0.02		
Cr		< 0.1			< 0.1		
Pb		< 0.2			< 0.2		
Zn		0.025	0.043	0.029	0.039	0.022	0.019
Hg		< 0.002			< 0.002		

Reductive substances in the aqueous extracts

Determination of the permanganate value of aqueous extracts from the medical devices provides the possibility of assessing the susceptibility of the polymeric material tested to oxidation processes proceeding in the course of accelerated ageing. A high content of reductive substances in the implant may bring about a risk of local reactivity after implantation.

Materials inert both in respect of chemistry and biology are sought in the designing of medical devices. An increase in the chemical reactivity of the implanted material promotes undesired biological processes which negatively affect the material itself by deteriorating its resistance to corrosion and cause local post-implantation effects.

Figure 4 presents the change in the reductive substance value (determined as permanganate oxidability) in aqueous extracts from prototype implants proceeding during accelerated ageing.

The permissible safety level of the permanganate value is marked in the graph. The permanganate value of aqueous extracts from the materials tested increased slightly for samples after 2 years of accelerated ageing. The values determined fall

Table 2. Changes proceeding in the amount of substances leachable in petroleum ether, isopropanol and water for the reference samples and implants prototypes before and after accelerated ageing.

Symbol of sample	Content of leachable substance, %									
	Extraction with petroleum ether				Extraction with 2-propanol				Extraction with H_2O	
	Time of extraction, h									
	2		1		1.5		1.5		0.5	
KO reference	0.408	0.330	0.121	0.129	0.009	< 0.001	<0.001	< 0.001	0.140	0.118
KO after 1 year of egeing	0.466	0.427	1.769	0.103	5.65	0.051	3.37	1.58	0.480	0.507
KO after 2 years of ageing	0.583	0.645	0.120	0.112	3.40	3.35	0.45	0.361	0.394	0.410
PF reference	0.276	0.358	0.132	0.174	< 0.001	0.038	< 0.001	< 0.001	0.087	0.077
PF after 1 year of egeing	0.392	0.420	0.092	0.095	0.154	0.174	0.077	0.091	0.102	0.090
PF after 2 years of ageing	0.463	0.439	0.222	0.216	1.46	1.54	0.275	0.183	0.479	0.520

fairly below the level regarded as safe in medical devices i.e. < 0.1 mg O_2/g.

Content of heavy metal ions in the aqueous extracts

In ***Table 1*** a quantitative profile is shown of the heavy metal content in aqueous extracts from the reference samples and prototype implants KO and PF before and after accelerated ageing.

In all of the samples of the prototype textile implants tested, the level of heavy metal content determined: Cd, Pb, Cr & Hg was below the limit of detection of the analytical method applied. The Zn content falls below the maximum permissible value both in the starting material and prototype textile implants subjected to accelerated ageing. The last parameter slightly declined in the course of accelerated ageing, which probably results from the fluctuating zinc content in the charge of the prototype knitted implants than from the ageing process itself.

Exhaustive extraction

Three solvents were used in the exhaustive extraction process: petroleum ether, 2-propanol and water. Changes proceeding in the amount of substances leachable in petroleum ether, 2-propanol and water for the reference samples and implants prototypes before and after the accelerated ageing are presented in ***Table 2***.

Testing to determine the amount of leachable substances was twice repeated.

In the accelerated ageing process an increased amount of petroleum ether-leachable substance could be observed extracted from the prototypes of textile PF products (1st extraction step).

In comparison to the initial sample, an increase of about 21% on average in the petroleum ether-leachable substance from the PF material was noted after accelerated ageing in conditions corresponding to 1 year of storage in real time. After the second extraction step, the amount of substance leached increased by 12% on average compared to the starting sample.

In the implants unmodified with low-temperature plasma, the change tendency was similar, although the amount of leached substance was higher.

After 2-year's accelerated ageing, the content of petroleum ether-leachable substance increased in the first step by about 66% on average compared to the initial sample and by 7% on average after the second step. The parameter value increased in total by 48% on average compared to the initial sample. A similar tendency appeared in the unmodified implants KO. However, the amount of leachable substances was distinctly higher.

A similar phenomenon was observed in the consecutive extraction steps made with isopropanol and water. The results confirm a higher chemical purity of knitted PF implants modified by low-temperature plasma treatment and their higher resistance to external factors in the course of accelerated ageing.

The pretreatment of fibre in the process of fibre surface modification by low-temperature plasma in the presence of low-molecular fluoroorganic compounds [18, 23] positively affects the chemical purity of the final medical device.

It ensues from literature [20] and Polish Pharmacopeia that the permissible amount of the substance leachable to the organic phase must not exceed 2 wt%. The quantitative composition of the extract is also important with respect to the substance posing a toxicity hazard. Since identification of the quantitative composition is difficult, the examination of biocompatibility according to Standard PN-EN ISO 10993-1 appears crucial for safe use of the devices. It ultimately verifies and documents the chemical and biological safety of prototypes of the medical devices designed.

Conclusions

The investigation in the accelerated ageing of ultra-light PP implants subjected to low-temperature plasma treatment with potential use in urogynecology and general surgery in the aspect of chemical purity constitutes a basis for determination of the effectiveness of the purifying process (pretreatment of accelerated ageing), the impact of the process upon the stability of chemical parameters and for defining changes proceeding in the course of storage in relation to real time.

It was found that the profiles of leachable substances for the KO and PF do not differ substantially, with values indicating high chemical purity.

The differences in chemical purity parameters, particularly those in exhaustive extraction, may be ascribed to the pretreatment made in the low-temperature plasma modification of the textile implant's surface.

Assessment of chemical purity parameters of the medical devices has an important function in the relating of the profile of leachable substances to the effects of the biocompatibility examination both in a local and systemic aspect. This will be assessed in future investigations devoted to biocompatibility according to directives of Standard PN-EN ISO 10993-1:2010 and in the examination of the impact of storage conditions upon the profile of the chemical purity of selected textile implants.

The results of the present research indicate that in the assessment of the profile of a leachable substance, advantage can be taken of some aspects of the chemical purity of textile medical devices, which

may differ depending upon the implant composition, its structure and processing, and the nature and time of contact with a human organism. Another aspect is also the assessment of the structural and topographic properties of the medical devices designed, permitting complex estimation of the impact of the said parameters upon the biological response to the textile device implanted.

Summary and conclusions

The research was aimed to:

1. estimate the impact of simulated storage conditions on chemical purity of tested polypropylene implant materials
2. assess the risk which quality impaired by long storage may bear upon useful properties of the implant materials.

Assessment of the storage conditions in real time is, for obvious reasons, not acceptable, which is why simulated accelerated storage was adopted, with elevated temperature playing the role of the time-accelerating factor according to the Arrhenius equation, which relates temperature with the reaction rate. 28 and 56 days were adopted as equivalents of 1 and 2 years of storage time.

Chemical purity represented by pH, the turbidity of extracts, the contents of chloride anions, the reductive substance (oxidability) and heavy metals, and the amount of leachable substance was tested for the freshly prepared materials and after the two (28 and 56 days) simulated periods.

The results are positive and encouraging. Most of the quality parameters did not change remarkably even after the 2-year period simulated. Practically all the quality indices remained in the permissible limit, giving proof that the storage time in terms of chemical purity does not pose a high risk for the PP implants tested.

An undesired increase in the leachable substance was noted in a few test results, although the majority remained positive. In the last analysis, the plasma- treated material, denoted as PF, proved to be a much better material, with low leached amounts of the substance. It may be concluded that the PF material carries a much lower risk than the untreated PP device.

All the results are comparable with test results after 1 and 2 years of real time storage.

The test results obtained indicate acceptable chemical purity and predict a low risk in clinical practice of the devices tested, the PF material in particular.

Assessment of chemical purity parameters of the medical devices has an important function in the relating of the profile of leachable substances to the effects of the biocompatibility examination both in a local and systemic aspect. Biocompatibility will be assessed in future investigations according to directives of Standard PN-EN ISO 10993-1:2010. It will provide final confirmation for surgical practice with a final assessment of the related risk of use after long storage.

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