Impact of Compaction Treatments on PET Yarns Used for Vascular Prostheses Manufacturing

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**ABSTRACT**

Textile cardiovascular prostheses are tubular structures made of polyester filaments. Woven prostheses are naturally tight but knitted ones are porous and can involve blood haemorrhage. Although compaction is necessary to reduce water permeability of knitted prostheses used for arterial replacement, it can induce degradation of mechanical properties of poly(ethylene terephthalate) (PET) yarns. We have studied the effect of chemical and thermal compaction process parameters on the physical and mechanical properties of PET yarns used for vascular prostheses manufacturing in order to find compaction systems that do not provoke important changes in the material characteristics. After chemical and thermal compactions, all yarn samples displayed longitudinal shrinkage accompanied by lateral swelling and a loss of mechanical properties. The analysis of chemical compaction results showed that fibre shrinkage and swelling are linked to the sizes of solvents' molecules and immersion duration. Compaction performances in thermal process are widely linked to treatment temperature. The effect of the two compaction treatments on a PET single jersey fabric permeability was studied as well.

**INTRODUCTION**

Graft implantation is today a common surgical procedure in the management of patients having severe blood circulation difficulties. Textile technology has provided several solutions for vascular surgery and a large number of textile vascular prostheses have been implanted in patients to revascularize districts downstream from diseased or injured arteries. Since 1954, the date of the first transplant on man, textile implants have saved millions of people, but they are not yet perfect because of the complexity of arterial biology and textile mechanics. Biocompatibility has been achieved but the problems of compliance and resistance to the blood flow still remain. In previous studies [1-3], we established correlations between porosity, flow nature and pathologies in prostheses such as thrombosis and hyperplasia. These pathologies are linked to the porosity of the textile...
structure because endothelial cells proliferation and blood particles deposit are conditioned by interstitial spaces inside prosthesis fabric.

Vascular prostheses made of poly(ethylene terephthalate) (PET) filaments have been largely employed for the replacement of large and medium injured or diseased arteries. This polymer, stabilized by high crystallinity is considered to be biostable.

Nevertheless, there have been several reports of long-term deterioration of devices in the human body, owing to fibres breakage and device dilatation. These failures have been attributed to structural defects, handling procedures and alterations of the polymer and the textile structures caused by post-manufacturing techniques employed for vascular prostheses [4-6].

One of the important post-manufacturing treatments employed for vascular prostheses made of PET is the compaction. The role of this treatment is to reduce water permeability of the textile structure used for the implant to an acceptable level, in order to avoid haemorrhage at the time of surgery and to maintain a viable vessel after implantation by allowing proper tissue growth for full-wall healing of the vessel. Compaction is necessary especially for knitted structures because of their initial permeability to liquids. Natural polyelectrolytes (alginate, collagen, chitosan, different glycosaminoglycans) and synthetic polyelectrolytes could be used to coat and make the textile structure impervious [7]. However, these proposals should be rigorously evaluated in-vivo because of the risks of inflammatory reactions as well as immunological responses against components of the implant that could be responsible for clinical complications following implantation [8].

Compaction can be accomplished by either thermal or chemical treatment. These treatments reverse the changes occurred during the heat drawing of the filament yarns. The chemical and thermal compaction systems use solvents or swelling agents in an aqueous medium for the former and dry or wet heat, for the latter under controlled conditions of temperature and duration of treatment. These compaction treatments lead to the swelling of PET fibres, and consequently, the reduction of interstitial vacuums within knitting which results in a decrease of water permeability [9-11].

In the literature, few research works have conducted studies on the compaction of a PET vascular prosthesis. Most of these studies were performed in the eighties. It has been reported, for example, that chemical compaction treatments result in significant changes in the performance of PET yarns used in vascular grafts [12]. It is therefore suggested that the failure of these materials may be in part due to alterations of the polymer and the fabric structures caused by these treatments. Documentation concerning thermal compaction is extremely rare. Physical parameters and materials used during industrial compaction processes are unknown and generally kept secret by few vascular prostheses manufacturers. In previous [13-15] studies, important modifications of the physicochemical properties of PET filaments extracted from explanted prostheses after ageing in the human body have been observed. These changes have been related to compaction treatments generally performed at high temperature and using aggressive chemicals.

The aim of this study is to understand and quantify the effect of chemical and thermal compaction processes on mechanical properties of PET yarns. This would allow determination of the best experimental conditions of compaction causing less degradation of the polymer.

**EXPERIMENTALS**

**Materials and Methods**

We have used textured multifilaments yarns made of biocompatible PET of 110 dtex and composed of 34 filaments having a circular section and a 20 μm diameter. These samples are conditioned in the testing laboratory under standard atmospheric conditions of 20 ± 1°C and 65 ± 2% R.H. for over 24 h according to French standard NFG 07-003. The effect of several compaction solutions and immersion time was evaluated on PET yarns. Five compaction solutions were utilized as described in Table 1.

For the chemical compaction treatments, we have worked at ambient temperature (20°C) as the first value for our tests. The temperature of vitreous transition (Tg) of poly(ethylene terephthalate) is located at approximately 70°C. We have chosen 75°C as the second value of temperature for the following tests.
Preliminary tests with the five compaction solutions for various immersion times showed that for both temperatures, 20°C and 75°C, longitudinal shrinkage remains constant between 0.5 and 5 min, then continues to increase until 10 min. So we have chosen the immersion times: 0.5 and 10 min for the tests.

The thermal approach is based on the use of dry heat [9-11]. We carried out an annealing treatment on a PET yarn by varying temperature and duration of the treatment. Five values of temperature located between 110°C and 180°C were selected. The thermal treatment was obtained by thermofixing samples in a "MATHIS" fixing stenter, equipped with an automatic heating system.

The effects of compaction systems on PET yarns were determined by measuring yarn geometry and tensile properties. These parameters were determined by testing ten specimens from each sample. Measurements of the longitudinal shrinkage rate for the treated PET yarns were taken according to NFG 07-101 standard for textured yarns under the standard conditions. An examination of fibres by projection microscope (Projectina®) allowed determination of PET fibres diameter. These measurements are in conformity with standard NF G 07-004. A magnification of 400 times was used. The longitudinal tensile tests on yarns were carried up on a traction testing machine according to the French standard NFG 07-302. The results were expressed as average tenacity (cN/tex) and elongation-at-break (%).

In order to investigate the impact of compaction treatment on the permeability of knitted prostheses, we performed chemical and thermal compaction treatments with a single jersey fabric made with the PET yarn. For samples knitting, a single jersey knitting machine was used. For this purpose, we developed an experimental device according to the recommendations of the Association for the Advancement of Medical Instrumentation (AAMI) [16] and the International Organisation for Standardization (ISO) [17]. The permeability test device (Figure 1) is composed of an upper tank permitting water flow through a circular knitted sample under a hydrostatic pressure of 120 mmHg. The flow rate through the knitted sample is measured by a flow meter (McMillan Co, model 101-6) and water permeability (mL/min/cm²) is calculated as follows:

$$ W = \frac{\text{Flow rate (mL/min)}}{\text{Sample area (cm}^2\text{)}} $$

<table>
<thead>
<tr>
<th>Compaction solution</th>
<th>Chemical product</th>
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<tbody>
<tr>
<td>S1</td>
<td>Methylene chloride</td>
</tr>
<tr>
<td>S2</td>
<td>Chloroform</td>
</tr>
<tr>
<td>S3</td>
<td>Dichloroethane</td>
</tr>
<tr>
<td>S4</td>
<td>94% Methylene chloride + 6% trichloroacetic acid</td>
</tr>
<tr>
<td>S5</td>
<td>94% Chloroform + 6% trichloroacetic acid</td>
</tr>
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Table 1. Chemical compaction solutions.
RESULTS AND DISCUSSION

Effect of Compaction Treatments on the Lateral Swelling and Longitudinal Shrinkage of Yarns

After chemical and thermal compactions, all yarns samples showed longitudinal shrinkage and lateral swelling. When the solvent is absorbed by the fibre, chemical intermolecular linkages (polymer-polymer) are replaced by solvent-polymer linkages. The molecular chains move away one from the other, become more independent and lose their cohesion with close chains and the free volume within material decreases. The absorbance of the solvent by the fibre is possible only when enough energy is supplied. This means that solvent is absorbed when polymer-solvent interactions are more favourable than polymer-polymer interactions [18].

The evolution of longitudinal shrinkage and lateral swelling corresponding to the five compaction solutions are given in Figure 2. We can note that transverse swelling and longitudinal shrinkage vary in the same way with compaction parameters (chemical agent types, immersion time and temperature). The compaction with the chloroform solution gives the highest swelling and shrinkage rates of yarns. Solutions 1 and 4 which contain a large volume of methylene chloride provoke higher swelling than that of the solution 3 which contains dichloroethane.

The difference between the results obtained from different solutions is related to the molecular sizes of solvents. Methylene chloride is characterized by small molecular sizes compared to the dichloroethane molecules (Table 2). It diffuses more easily inside fibre compared to high molecular size products. The addition of trichloroacetic acid to the chloroform also contributes to substantial swelling. When we increase the duration of immersion, the chemical product, even if it has an important size, has enough time to diffuse into the polymer structure. For this reason, values of longitudinal shrinkage rate and transverse swelling rate obtained for an immersion time of 10 min are generally superior to those obtained after a treatment of 0.5 min.

The glass transition temperature ($T_g$) marks the onset of segmental mobility of the polymer. It is the temperature below which the polymer segments do not have sufficient energy to move past one another. At a temperature of 75°C, which corresponds to the glass transition zone, the polymer molecules in the amorphous domains have sufficient mobility to increase the ability of the solvent to diffuse into the polymer [19]. Therefore, it appears clearly that, for the same immersion time, sample yarns treated with the different compaction solutions at this temperature will undergo a greater effect of longitudinal shrinkage and lateral swelling compared to yarns treated at 20°C.

Table 2. Molar weight of chemical products used for various compaction solutions.

<table>
<thead>
<tr>
<th>Chemical product</th>
<th>Molar weight (g/mol)</th>
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</thead>
<tbody>
<tr>
<td>Methylene chloride</td>
<td>CH$_2$Cl$_2$</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>CICH$_2$-CH$_2$Cl</td>
</tr>
<tr>
<td>Trichloroacetic acid</td>
<td>Cl$_3$COOH</td>
</tr>
<tr>
<td>Chloroform</td>
<td>CHCl$_3$</td>
</tr>
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</table>
In the literature, it was reported that the mobility of the molecular structure of the PET permits diameter increase and longitudinal shrinkage \([9]\). Shrinkage in fibres is usually attributed to the rubber-like retractive forces present in the amorphous regions of the commercial fibres and to the chain folding effect \([20-22]\). Another factor governing the yarn shrinkage is the orientation of the molecular segments in these amorphous domains \([16]\). The molecular orientation of the non-crystalline domains slows down the solvent diffusion \([23,24]\). The diffusion rate of solvent molecules into the fibre matrix depends on the amount of non-crystalline regions present \([25]\).

The thermal compaction treatments were obtained by yarn heat setting during 1 and 10 min. The percentage of longitudinal shrinkage and lateral swelling rates for the five annealed samples is shown as a function of heat-setting temperatures for different durations of heat setting in Figure 3. The curves indicate that longitudinal shrinkage and lateral swelling may be considered as a thermally activated process.

For thermal treatment, the longitudinal shrinkage increases with temperature. The same variations were obtained with the lateral swelling. The temperature has a more important effect on the transverse swelling and the longitudinal shrinking rates than treatment duration. We note that at high heat-settings temperatures, the time of heat-setting has a relatively large effect on swelling. Gacen et al. have shown that crystallinity increases with the temperature and/or the annealing time, with a high effect of temperature \([26]\).

The values of longitudinal shrinkage rate obtained for the temperatures 110°C and 120°C for the two annealing durations are situated between 5 and 6.5%. For the temperatures 140°C, 155°C and 180°C, the values of longitudinal shrinkage rate vary from 11% to 24%. The thermal treatment at 180°C during 1 and 10 min gives the most interesting values of lateral swelling and longitudinal shrinkage (28% and 31%, respectively). The obtained results at 155°C and 1 min correspond to the maximal value obtained by the chemical compaction and which corresponds to a treatment with the solution 2 at 75°C during 10 min. Thermal treatment at 140°C during 10 min and the chemical treatment with solution 4 at 75°C give similar results.

**Effect of Compaction Treatments on Tenacity and Elongation-at-break of the PET Yarns**

Figures 4 and 5 show that chemical and thermal compactions induce significant changes in the break load and elongation of the yarns. In the literature it has been shown that swelling by chemical agents causes chain scission, loss of crystalline orientation and break of intermolecular bonds \([27]\). This would justify the decrease of tensile performances and the increase of elongation-at-break. Gupta et al. \([20]\) have also shown that there is a considerable shrinkage in free-annealed samples. Due to this shrinkage, the orientation of the molecules is reduced. When these fibres are subjected to a tensile test, they extend more than the untreated fibres. Compaction treatment involves also a reduction of yarn modulus. The textile structures deform more easily after compaction. This phenomenon has been also reported in literature \([28]\) and it is associated to fibre fatigue. The increase of elongation for PET yarns used for vascular prostheses can cause major mechanical problems. Theses problems are mainly aneurysms which are long-term dilation of the graft (balloon-like bulge) that causes
serious blood flow complications [2,3]. According to Dieval et al., the manufacturing process of the prostheses modulates graft ageing by modification of the macromolecular structure of the polymer. The different treatments undergone by prosthesis, involve the appearance of permanent and heterogeneous tensions in the knitted structure and consequently localized degradations could be obtained. The presence of a solvent due to compaction process can also accelerate the formation of polymer splits or cracks especially when the graft is under external tension [29].

Compaction with methylene chloride (solution 1) at 75°C for immersion time 0.5 and 10 min leads to important reductions in tenacity as shown in Figure 4 (42% and 45%). The treatment at 20°C during 10 min gives a reduction in tenacity around 33%. The treatment at 75°C during 10 min with the chloroform solution (solution 2) led to the best rate of lateral swelling and longitudinal shrinkage but a reduction of tenacity of 44% and an increase in elongation of 48% are obtained. For the dichloroethane solution (solution 3), the treatment realized at 75°C during 0.5 min involved a reduction in tenacity of about 39% and 41% for an immersion time of 10 min. The second best rates of lateral swelling and longitudinal shrinkage were obtained with a 94% methylene chloride and 6% trichloroacetic acid solution (solution 4) for a treatment at 75°C during 10 min. This treatment gives a notable increase in elongation (65%) and a reduction of 44% in tenacity. A treatment at 20°C during 10 min with the solution 5 which contains 94% chloroform and 6% trichloroacetic acid (solution 5), gives a reduction in tenacity of 43%.

For the thermal treatments, the increase in the temperature involved an increase of the rate of elongation-at-break and tenacity loss of the PET yarns as shown in Figure 5. For low temperatures, the duration of the treatment has a low influence on the tenacity values. The elongation-at-break of the yarn samples increases with temperature because of the relaxation
of internal tensions inside fibres under the heat effect. When temperature is above the $T_g$, the polymer segments rearrange to relieve the external stress. This rearrangement due to the mobility of polymer macromolecular chains permits the increase of elongation before break.

For the same values of longitudinal shrinkage and lateral swelling, some compaction processes can cause more degradation on the poly(ethylene terephthalate) fibres than others. For example, the yarns treated with the methylene chloride solution (S1) at 20°C during 10 min have a lateral swelling and a longitudinal shrinkage of 6%, with a loss of 34% in tenacity. Conversely, the samples yarn treated with the solution S5 at 75°C during 0.5 min show the same values of lateral swelling and longitudinal shrinkage, but with a loss of 44% in tenacity.

Chemical compaction with solution 1 at 20°C, 10 min and thermal compaction at 120°C, 10 min showed similar fibre shrinkages and swellings but different losses in tenacity (respectively, 33% and 43%).

**Effect of Compaction Treatments on Vascular Prostheses Permeability**

Figure 6 shows the impact of chemical and thermal compactions on prostheses permeability. Permeability decreases after chemical treatment. Solution 2 showed the highest and solution 3 showed the lowest impact on fabric permeability. According to Figures 2 and 6 polyester fibre shrinkage and swelling reduce the knitted structure permeability. After compaction yarn shrinks and yarn length forming the knitted loop is shorter, consequently fabric stitches are denser and fabric has lower permeability.

The curve showing the influence of thermal compaction temperature on the prosthesis permeability curve indicates that increasing fixing stenter temperature reduces prosthesis permeability. Higher temperatures give higher fibre shrinkage and swelling and gives better compaction results. This is due to shrinkage and swelling phenomena observed with PET fibres. High temperature enables molecular chains to move easily and fibre to shrink and swell. The knitted structure is consequently less porous.

**CONCLUSION**

The aim of this study was to compare the effect of thermal and chemical compaction systems on the mechanical properties of poly(ethylene terephthalate) yarns in order to find the compaction system that induces the lowest reduction of the mechanical performances of the material. We have demonstrated that all compaction systems caused losses of the mechanical properties of the polyester yarns. After chemical or thermal compactions, all yarns samples showed longitudinal shrinkage and lateral swelling linked to loss of crystalline orientation, chain scission and broken intermolecular bonds. It can be concluded that compaction with methylene chloride during 0.5 min at 20°C gives interesting results for the tenacity decrease, but do not induce very interesting swelling...
and shrinkage results. At 75°C, the swelling and shrinkage effects are more important but tenacity reduction reaches 42%. Compaction with chloroform gives a better swelling of fibres at 75°C for 10 min, but it generates a substantial decrease of the yarn tenacity.

The choice of the compaction process depends largely on the properties of the initial knitted textile structure used for vascular prosthesis manufacturing. In order to have the minimum degradation of the mechanical properties of the PET yarns forming these prostheses, it would be necessary to use an initially tight knitted structure.

REFERENCES

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