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Processing of Chitosan Yarn into Knitted Fabrics

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Abstract
This article focuses on a preliminary investigation into the preparation of knitted fabrics from a 300-filament chitosan yarn containing nanoparticles of silver, platinum, copper and gold. The chitosan yarn was assessed with respect to its suitability for the preparation of knitted fabrics on a numerically controlled flat bed knitting machine. Estimated were mechanical and sorption properties, apparent density, thickness and air permeability of knitted fabrics made up of chitosan yarn functionalised by nanoparticles.

Key words: *chitosan yarn, nanoparticles, chitosan knitted fabric, mechanical properties.*

Introduction

Derivatives of chitin like chitosan and dibutyrylochitin find uses in new generations of biomaterials. Fibrous forms of polymers like nano- and microfibres, staple fibres and yarns [1 - 3] have been used widely in several application domains especially in medicine. These fibrous forms are frequently modified or functionalized by the addition of multiwall carbon nanotubes, nanoparticles of various metals, calcium phosphate, collagen, fibroin or keratin [4 - 11]. At the Institute of Biopolymers and Chemical Fibres (IBWCh), Łódź, Poland, a method was developed to produce multifilament chitosan yarn [3]. The material was applied in the preparation of semi-absorbable surgical meshes and prostheses of nerves [3, 12].

Chitosan-metal complexes reveal an enhanced antibacterial activity when compared with virgin chitosan; the chitosan-copper complex exerts anti-tumor properties. Silver nanoparticles penetrate the walls of *Escherichia coli* and *Staphylococcus aureus* cells and appear to be more effective than antibiotics. Nanoparticles of silver and copper are similarly effective in inhibiting the growth of bacteria, which might have a better effect if carbon or polymers underpin the metals' activity [13 - 16]. Pure chitosan yarns have been applied in the construction of knitted fabrics for uses in textile scaffolds [17 - 21].

The main objective of the work is to assess the suitability of chitosan fibres functionalized by nanoparticles in the preparation of knitted fabrics with a view to their potential use in dressings and scaffolds. For investigation purposes, a 300-filament chitosan yarn was prepared with the addition of nanoparticles: silver, gold, copper and platinum (***Table 1***). An assessment of the antibacterial activity of the nanoparticle-functionalized chitosan yarn was conducted aimed at preparing knitted fabrics by a flat bed knitting machine. The knitted fabrics were characterised by tenacity and extension, sorption properties, apparent density, surface density, thickness, and air permeability.

Experimental

Materials

300-filament nanoparticles-modified chitosan yarn with linear density of 900 dtex was prepared in the form of a continuous, untwisted yarn, collected on paper spools. The yarn was spun on a spinning line at IBWCh. The chitosan yarn obtained contained nanoparticles of silver, gold, copper and platinum. The nanoparticles were added to the chitosan spinning dope.

The following nanoparticles were used in the investigation:

- Silver nanoparticles - Hydrosilver 1000, supplied by Amepox Co, Poland. It contains nanoparticles stabilised with nanosilica.
- Gold, platinum and copper, supplied by nano-Tech Co, Poland, in the form of aqueous colloidal solutions with a nanoparticle concentration of 20 - 1000 mg/kg

Methods

Preparation of chitosan knitted fabrics

Chitosan yarn modified by nanoparticles was employed in the preparation of knitted fabrics on typical textile machinery at the Faculty of Material Technologies and Textile Design, Lodz University of Technology.

The knitted fabrics were prepared on a numerically controlled flat bed knitting machine type CMS 330.6 E12 by STOLL Co., using a satin twill braid (***Figure 1***).

Satin twill braid was used in order to reduce yarn tension in the knitting zone, to form consecutive stiches on one needle only, and to reduce the lead angle of the yarn on the sinkers, from 180° to 90°. To supply the yarn to the knitting zone of the machine, a special feeding system was employed comprising an electronic constant-stress feeder, a friction feed wheel and a special pneumatic compensation device. Parameters of the process were adopted in order to provide a stress-less processing of the yarn: knitting rate

Table 1. *Characteristics of chitosan yarn.*

Symbol of yarn	Concentration of nanoparticles, g/kg	Kind of nanoparticles
MFChit-S 1	53.8	silver
MFChit-S 2	9.2	gold
MFChit-S 3	0	no nanoparticles
MFChit-S 4	6.6	platinum
MFChit-S 5	57.3 (243 Ca)	silver (calcium)
MFChit-S 6	50.3 (320 Ca)	silver (calcium)
MFChit-S 7	6.6	copper

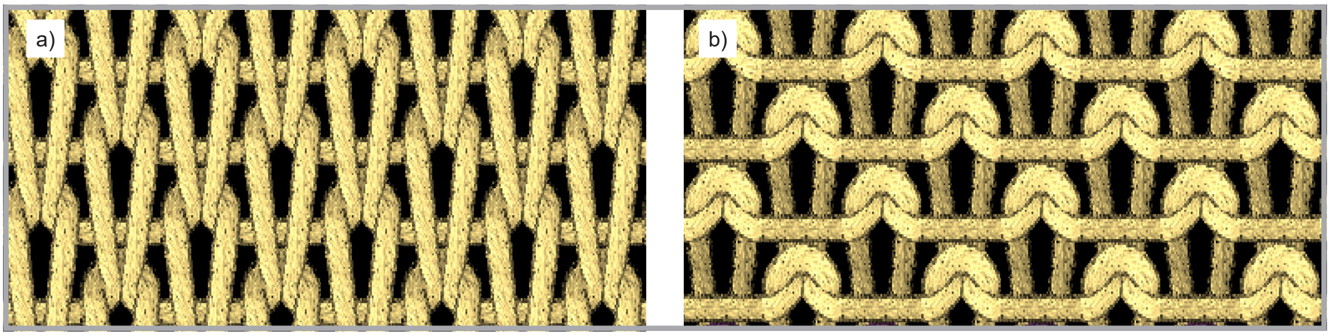

Figure 1. *Satin - weft knitted fabric, a – face side, b - backside.*

of 0.2 m/s, high sinking depth and minimal take-off speed. Seven various 300-filament yarns were prepared for knitting (***Table 1***).

Analytical methods

Estimation of fibre mechanical properties

Mechanical properties of the chitosan fibre were tested according to standards PN-ISO-1973:2011 and PN-EN ISO 5075:1999. Measurements were conducted at RH of 65 ± 4% and temperature of 20 ± 2 °C.

Estimation of molecular mass distribution (MMD) of chitosan in the yarn

The distribution of the molecular mass of chitosan in the yarn was determined by gel chromatography (GPC/SEC). The function of mass distribution (*MMD*), the average molecular mass ($\overline{M}_n$, $\overline{M}_w$) and polydispersity ($\overline{M}n/\overline{M}w$) were determined as described elsewhere [11]. The results were calculated using the universal calibration method with parameters in the Mark-Houwinek equation, amounting to: a = 0.625 & k = 62×10^{-5} ml/g for the PEO/PEG standards, and a = 0.76 & k = 74×10^{-5} ml/g for chitosan [22], respectively.

Antibacterial activity of chitosan yarn

The antibacterial activity of the Chitosan yarn against *Staphylococcus aureus* ATTC 6538 and *Escherichia coli,* was estimated using the quantitative test, according to Standard JIS L 1902:2002. The number of live bacteria growing in the fibre sample tested and in a standard substrate (regular chitosan fibre) was determined after 24-hours of incubation.

Estimation of calcium-, silver- and copper content in the yarn

The calcium content was determined directly in a mineralized sample by Flame Atomic Absorption Spectrometry (FAAS), at a wavelength of 213.9 nm. The fibre was first incinerated at 575 °C and then, mineralized in 70% HNO_3 in a microwave oven. A SCAN-1 atomic absorption spectrometer (Thermo Jarrell Ash Co., USA) was used for the analysis. The background was corrected by the Smith-Hiettje method [23].

The silver and copper content was determined directly in a mineralized sample by Flame Atomic Absorption Spectrometry (FAAS) at a wavelength of 328.1 nm. The fibre was first incinerated at 575 °C and then, mineralized in 75% HNO_3 in a microwave oven. A SCAN-1 atomic absorption spectrometer (Thermo Jarrell Ash Co.) was used in the analysis. The background was corrected using the Smith-Hiettje method [23].

The content of other nanoparticles in the fibre was not determined; content values are given as weighted.

Estimation of physical properties of the knitted fabrics

The thickness was measured by a thickness gauge - Arthur Meiber KG LTG, according to Standard PN-EN ISO 5084:1999. A pressure of 2 Pa was applied in the measurements. A surface of 1000 mm^2 was measured and 10 measurements made to compute an average value with (having an accuracy of 0.01 mm) the variation coefficient.

The surface mass was measured in accordance with the standard PN-EN 29073-1:1994. According to PN-EN ISO 186:2004, a sample of about 50,000 mm^2 was taken from the material tested. The samples were air-conditioned prior to measurement of the size and weight. The surface mass of the knitted fabrics was determined with an accuracy of up to 0.1% of the weighted mass.

The apparent density is the quotient of the mass of the textile material by its volume, including all voids appearing between the structural elements of the material. The apparent density was estimated in accordance with Standard PN-85/P-04688. Measurements were made at three points along the sample and at three points across, with an accuracy of 1 mm, and then the thickness of the material was determined according to Standard PN-EN ISO 5084:1999 and its mass with an accuracy of up to 0.2% of the weighted mass.

Air permeability was measured according to Standard PN-EN ISO 9237:1998 by means of Textest FX 3300 apparatus. Measurements were made at 10 points, at a distance of 5 cm from the edge and in a partial vacuum of 100 Pa for a nominal measurement surface of 20 cm^2.

The tenacity and extension of the knitted fabrics were measured on an Instron machine according to Standard PN-EN 29073-3:1994.

The samples were kept for 24 hours and air-conditioned at 25 °C and RH of 65%. The rate of the movement speed of the transversal bottom clamp was 100 mm/min, with a distance of 200 mm between the clamps, and 50 mm width of the sample. Samples were cut out along the wales and courses of the knitted fabrics.

The sorption of the knitted fabrics was estimated by means of a sorption meter -SORP 3. The textile materials were put into a special cup filled with water where the material imbibed water.

Results and discussion

Characteristic of chitosan yarn

Nanoparticles modified chitosan yarn was prepared on an experimental spinning line at IBWCh: 300-filament continuous yarn for the processing of knitted fabrics.

300-filament yarn containing nanoparticles of silver, copper and calcium, were prepared for the manufacture of knitted

Table 2. *Antibacterial activity of nanoparticles containing chitosan yarns against Staphylococcus aureus.*

Symbol of fibre	Kind of nanoparticles	Time, h	No of bacteria, jtk/pr	Bacteriostatic activity	Bactericidal activity	Growth
Chit-0	(standard)	0	3.5×10^4	-	-	-
		24	1.3×10^7	-	-	2.6
MFChit-S 1	silver		2.3×10^1	5.7	3.1	-
MFChit-S 2	gold		3.1×10^6	0.6	-2.0	-
MFChit-S 7	copper		1.0×10^7	0.1	-2.5	-

Table 3. *Antibacterial activity of nanoparticles containing chitosan yarns against Escherichia coli.*

Symbol of fibre	Kind of nanoparticles	Time, h	No of bacteria, jtk/pr	Bacteriostatic activity	Bactericidal activity	Growth
Chit-0	(standard)	0	1.6×10^5	-	-	-
		24	1.7×10^8	-	-	3.0
MFChit-S 1	silver	24	1.5×10^2	6.0	3.0	-
MFChit-S 2	gold		3.6×10^7	0.6	-2.4	-
MFChit-S 7	copper		4.1×10^7	0.6	-2.4	-

Table 4. *Mechanical properties of elemental fibers in chitosan yarn.*

Parameter		MFChit-S 3	MFChit-S 5	MFChit- S 6
		without nanoparticles	silver	silver
Linear density	dtex	3.37 ± 0.13	3.00 ± 0.11	4.21 ± 0.07
Breaking force of air conditioned fibre	cN	3.41 ± 0.13	4.11 ± 0.12	4.47 ± 0.21
Tenacity air conditioned	cN/tex	10.1	13.7	10.6
Elongation at break, air conditioned	%	11.0 ± 1.4	8.8 ± 0.8	10.2 ± 0.5
Breaking force, wet state	cN	0.733 ± 0.036	1.23 ± 0.04	1.24 ± 0.07
Tenacity, wet state	cN/tex	2.18	4.1	2.95
Elongation at break wet state	%	20 ± 2	20 ± 2	25 ± 2
Loop tenacity, air conditioned	cN/tex	2.24	3.23	2.92
Relative loop tenacity	%	22.1	23.6	27.5

Table 5. *Mechanical properties of chitosan yarns.*

Parameter		MFChit-S 3	MFChit-S 4	MFChit-S 5	MFChit-S 6
		without nanoparticles	platinum	silver	silver
Linear density of yarn	dtex	839 ± 13	853 ± 8	831 ± 1	579 ± 10
Breaking force of air conditioned	cN	850 ± 25	787 ± 31	980 ± 21	539 ± 25
Tenacity, air conditioned	cN/tex	10.1	9.2	11.8	9.3
Elongation at break air conditioned	%	3.79 ± 0.44	2.08 ± 0.21	3.54 ± 0.28	3.15 ± 0.54

fabrics (***Table 1***). Chitosan yarn denoted by MFChit-S 5 contains 57.3 mg/kg of silver and 243 mg/kg of calcium, while yarn MFChit-S 6 carries 50.3 mg/kg of silver and 320 mg/kg of calcium.

Bacteriostatic and bactericidal activity of chitosan yarn

Antibacterial activity was examined in selected chitosan yarns containing nanoparticles of gold, silver and copper, and in chitosan staple fibre with silver. Results are compiled in ***Table 2***.

Bacteriostatic and bactericidal activity against the strains of *Escherichia coli* & *Staphylococcus aureus* feature in chitosan yarn with a content of nanoparticles of silver above 50 mg/kg (***Table 2*** and ***3***). As earlier reported [7], chitosan fibres with a silver content are active against both Gram negative and Gram positive bacteria. Fibres with nanoparticles of gold and copper are neither bacteriostatic nor bactericidal to both Gram negative and Gram positive bacteria. The low content of copper (6.6 mg/kg) might have been the reason. The yarn with copper was stiff and brittle, and hence it was not qualified for use in knitting.

Mechanical properties of chitosan fibre

Mechanical properties of chitosan elemental fibres and yarn are shown in ***Tables 4*** and ***5***. Problems arose with yarn containing platinum (MFChit-S 4); attempts failed to separate elemental fibres from the yarn bundle (***Table 4***).

The tenacity of air conditioned elemental chitosan fibres is in the range of 10 - 14 cN/tex with a 10% elongation. The wet tenacity is 3 - 4 times lower and the wet elongation 2 times higher than in the air-conditioned fibres. The differences in mechanical properties can be explained by the variation in spinning conditions and by the kind of nanoparticles added.

The linear density was measured in order to compare the results with calculated values. The yarn linear density is a multiple of the elemental fibre measurement; it is at the level of 900 dtex, close to that of the value computed, except yarn denoted by MFChit-S 6. Yarn tenacity falls within the range of 9.2-12.0 cN/tex with an elongation of about 3%.

The relative error of elongation measurements is around 10%, while the absolute error falls within the range of 0.20% to 0.54%, witnessing a good uniformity of the yarn. Tenacity and elongation are lower in the yarn than in the elemental fibres. Yarns with the highest loop tenacity and elongation (MFChit-S 5, MFChit-S 6) seem to be suitable for knitting.

Molecular mass distribution (MMD) and polydispersity index (PDI) of chitosan yarns

Examined were: the MMD, medium value of molecular mass ($\overline{M}_n$, $\overline{M}_w$) and PDI in chitosan yarns (***Figure 2***, ***Table 6***).

The chitosan yarns analysed differ in molecular mass and polydispersity (PDI). (***Table 6***, ***Figure 2***), with the yarn denoted by MFChit-S 5 having the highest molecular mass M_w and the lowest PDI of 3.6 is featured by the higher tenacity. It is an indication that the material is suited for performing the knitting process.

Investigation into the preparation of chitosan knitted fabrics

Seven lots of chitosan 300-filament yarn were prepared for studying the knitting (***Table 1***). It appeared that the yarns have the drawbacks of high stiffness and brittleness and suffer from low resistance to friction on yarn guides as well as the hooking of needles with small dimensions. This was the reason why satin weft knitted fabric was adopted in the trials (explanation found in publications [24 - 27]). Positive results were achieved with four various chitosan yarns:

MFChit-S 1 (with silver nanoparticles) - the knitted fabric prepared was of medium quality. Broken ends of yarn on the bobbin caused disturbance of the knitting process which, in fact, disqualifies the yarn package for knitting (***Figure 3***). It calls for improving the spinning performance i.e. the building up of the packing on the bobbins, particularly eliminating the reason for disturbances in the knitting.

MFChit-S 2 (with gold nanoparticles) - yarn unsuited for knitting. The fabric prepared was faulty (***Figure 4***); its edges are much more prone to up-winding as result of yarn stiffness.

A lot of broken fibres appear on both the face and backside, being the reason behind many faults in the structure of the fabric.

MFChit-S 3 (without nanoparticles) - a knitted fabric of medium quality was prepared, although with a lot of broken fibres and faulty stiches caused by inadequate yarn quality. The yarn is stiff, with broken ends appearing in the packing. It altogether calls for improvement in the spinning and up- winding of the yarn packages.

A lot of broken fibres appear on both the face and backside, being the reason for many faults in the structure of the fabric.

MFChit-S 4 (platinum nanoparticles) - yarn unsuited for knitting. The fabric prepared is of low quality, with many fractures. The yarn was stiff and brittle, causing problems in knitting.

MFChit-S 5 (silver nanoparticles) - the prepared knitted fabric was of me-

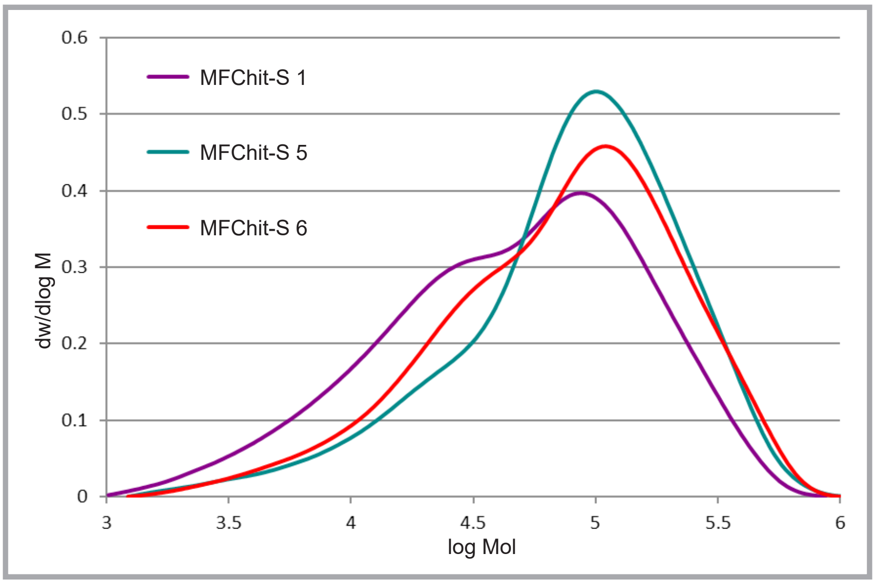

Figure 2. *Molecular mass distribution in chitosan yarn containing silver nanoparticles.*

Table 6. *MMD characteristics of chitosan yarn containing silver nanoparticles.*

Parameter	Symbol of 300-filament chitosan yarn		
	MFChit-S 1	MFChit-S 5	MFChit-S 6
Mp	93603	114494	102743
Mn	22131	34572	24362
Mw	82913	124858	100298
Mz	177402	251112	221664
Mz+1	272698	370576	337799
Mv	73841	112237	88666
Polydispersity PDI	3.747	3.612	4.117

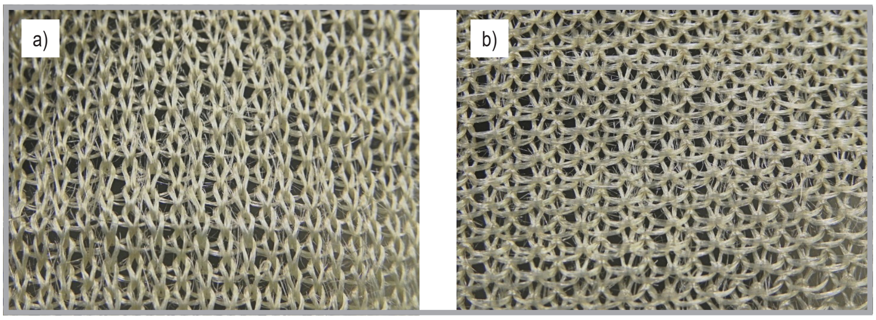

Figure 3. *Image of chitosan knitted fabric with content of silver nanoparticles - symbol MFChit-S 1, a - face side - a lot of fibers can be seen, broken in the course of knitting, b - reverse side - a lot of fibers can be seen, broken in the course of knitting.*

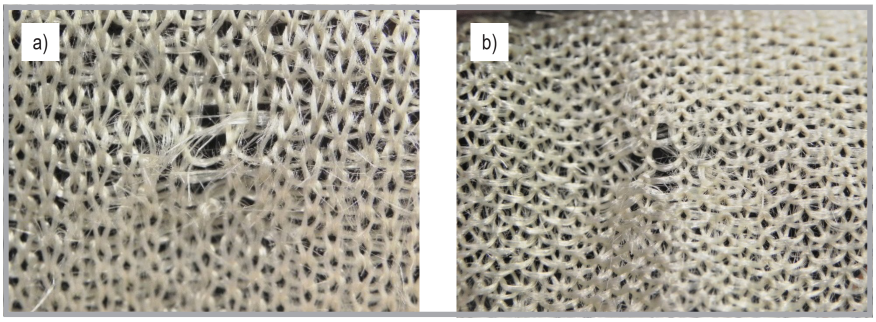

Figure 4. *Image of chitosan knitted fabric with a content of gold nanoparticles - marked MFChit-S 2, a - face side, b - backside.*

Figure 5. Image of chitosan knitted fabric marked MFChit-S 6, backside.

dium quality. Broken ends appear on the bobbin calling for improvement of the yarn to continue knitting trials.

MFChit-S 6 (silver nanoparticles) - the yarn is suited for knitting; a fabric with good quality was prepared (***Figure 5***).

The images in ***Figure 5*** present longitudinal view of the fabric surface, where protruding fibres can be seen. Such fibres deteriorate the quality of the fabric, the strength in particular.

The bobbins with 300-filament chitosan yarn revealed disturbances in the yarn's package pattern, which is a deficiency that causes problems in the unwinding of the yarn from the bobbin. Another fault was the broken, unbounded ends of the yarn, which badly affects the knitting process, causing repeated threading and splicing of broken ends and knitting knots into the fabric, which may lead to the disruption of the knot-holding stitch. The high stiffness of the yarn limits the proper forming of the stitches.

Yarns marked: MFChit-S 2 and MFChit-S 4 are quality-wise, unsuitable for knitting.
Knitted fabrics with reasonable quality were made from the following yarns:
- MFChit-S 1: fabric with a lot of broken fibres
- MFChit-S 5: fabric with fewer broken fibres than in the MFChit-S 1 and MFChit-S 2 variants
- MFChit-S 6: quality-wise is the best fabric amongst all variants investigated.

The positive attempts at preparing chitosan knitted fabrics augur the chance of their manufacture on knitting machinery, which may be regarded as an achievement since it is chitosan knitwear that was made from pure chitosan yarn.

Physical properties of chitosan knitted fabrics

Four various knitted fabrics were examined for their physical properties (***Table 7***).

Table 7. *Physical properties of chitosan knitted fabrics.*

Kind of fabric	Thickness, mm	Standard deviation, mm	Variance, %	M_p, g/m^2	Apparent density, kg/m^3	Air-permeability, l/m^2/s
MFChit-S 1	1.38	0.02	0.05	262.31	190.26	5210
MFChit-S 3	1.72	0.02	0.05	287.86	167.43	5360
MFChit-S 5	1.15	0.01	0.02	285.44	248.21	4380
MFChit-S 6	0.76	0.01	0.02	178.00	234.21	4210

Table 8. *Tear resistance and elongation of the knitted fabrics.*

Kind of fabric		Elongation at break, mm	Breaking force, N	Young modulus, MPa
MFChit-S 1	wale	76.88	50.77	4.14
	course	99.20	68.94	3.71
MFChit-S 3	wale	75.18	47.59	2.60
	course	104.70	51.10	0.89
MFChit-S 5	wale	85.31	122.20	12.92
	course	135.10	104.20	2.30
MFChit-S 6	wale	85.79	68.95	13.57
	course	127.40	74.26	4.73

Table 9. *Sorption capacity of the chitosan knitted fabrics.*

Kind of fabric	Sorption capacity, g/g	Standard deviation, g/g
MFChit-S 1	7.34	0.32
MFChit-S 3	6.80	0.28
MFChit-S 5	7.71	0.43
MFChit-S 6	8.69	1.53

The highest thickness was found in the fabric made of chitosan yarn MFChit-S 3, while MFChit-S 6 produced the thinnest fabric. Probably the reason for differences in the thickness of the fabrics was the linear density of the yarn. Air permeability is closely related to the apparent density of the fabrics, which goes down with an increasing surface mass.

Fabrics marked MFChit-S 5 drawn along wales and courses, demonstrate better tear resistance in contrast to MFChit-S 1 fabrics, with the lowest value (***Table 8***).

Drawing along the courses produces much better tear resistance than along the wales. The reason for such different tear resistance of the knitted fabrics was the kind of chitosan yarn.

The sorption capacity of the chitosan knitted fabrics were at the level 7 g/g. The highest sorption was found in the fabric made of t yarn marked MFChit-S 6 and the lowest value in the fabric of yarn MFChit-S 3 (***Table 9***, ***Figure 6***).

Conclusion

Chitosan yarn modified with nanoparticles of various metals were assessed in regard to their suitability for the manufacture of knitted fabrics using typical industrial equipment. The addition of gold, copper and platinum in nano- form also gave rise to quality deterioration of the chitosan fibres. Consequently yarn with these metals could not be qualified as suitable material for textile processing. Nanoparticles of silver proved to be the right additive for chitosan fibres, conferring enhanced bacteriostatic and bactericidal properties upon them. Yarn bearing such properties is a promising material for medical use like dressings, scaffolds, and surgery meshes. To achieve this purpose, it needs to be processed into textile forms, like knitted fabrics. Attempts to prepare knitted materials from chitosan yarn containing nanoparticles of silver produced positive results. However, it must be admitted that the processes did not run smoothly, thereby revealing seriously inadequate properties of the processed fibres, like low tenacity and elongation, poor quality of the yarn packages and high static electricity on the fibre, which, in consequence, led to defects in the textile materials prepared. Such deficiencies must be overcome and the quality of the yarn should be substantially

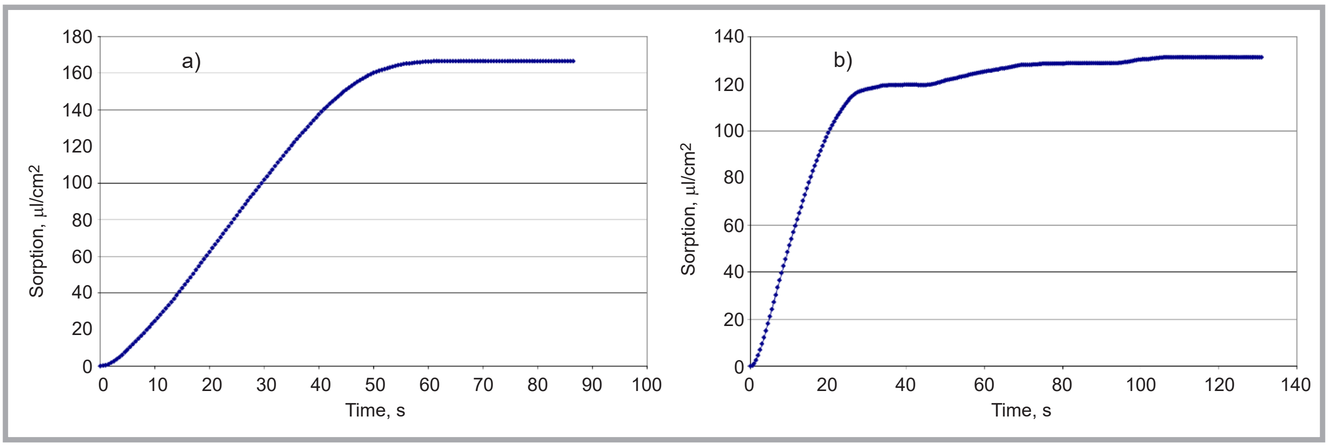

Figure 6. *Sorption of chitosan knitted fabric MFChit-S 3 (a) and MFChit-S 6 (b) with content of silver nanoparticles.*

corrected in additional research concerned with the spinning of chitosan yarn.

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