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Multifunctional Alginate Fibres with Anti-bacterial Properties

Abstract
Conditions for the manufacture of fibres designed for medical applications from zinc alginate and copper alginate have been developed. The high moisture absorption and anti-bacterial effects of fibres from zinc or copper alginate will allow the production of a new generation of dressing materials. At the same time, the tenacity of copper alginate fibres at a level of 21.41 cN/tex and their modified electric properties will make it possible to obtain flat textile materials designed for medical application (hospital linen, compression bandages). A comparative analysis has been carried out to assess the effects of fibre spinning conditions on the porous structure, moisture absorption and strength properties of fibres from zinc and copper alginates.

Key words: *alginate fibres, zinc alginate, copper alginate, anti-bacterial properties, medical applications, as-spun draw ratio, draw ratio, tenacity, absorption, water retention, pores.*

Introduction

The developmental revival of alginate fibres is due to the medical applications of these fibres, mainly for the manufacture of modern, active dressing materials [1,2]. This development has been facilitated by the following factors:

- inexpensive, easily available natural raw materials such as sea-weeds or algae,
- the specific properties of alginates which facilitate the healing of wounds,
- the high moisture absorption and ion-exchange capabilities of alginate fibres,
- the perfect biodegradability of the fibre-forming material.

The alginate dressings currently available on the market have mainly been manufactured from fibres of calcium alginate or sodium-calcium alginate[3]. Depending on the quantity of the mannuric or guluronic acid derivatives in the fibre-forming material, the fibres change into gel form to varying degrees [4,5]. On the other hand, sodium alginate fibres are capable of changing completely into gel form [6].

Modern active dressings, besides their function of providing a moist wound environment, should also be adapted to the stage of wound healing and, on this basis, capable of stimulating the granulating process or protecting against the damage of a newly formed tissue [7,8].

An additional advantage of alginate fibres is that they are relatively easy to modify by incorporating appropriate metal ions, microelements or other biologically active substances that accelerate the healing process or have bacteriostatic properties [9].

The replacement of sodium ions with zinc or copper ions during the solidification stage will allow zinc alginate or copper alginate fibres with suitable properties for medical applications to be prepared.

Zinc alginate fibres, in addition to their features resulting from the composition of fibre-forming material and consisting in accelerating the wound healing process, show a high moisture absorption and antibacterial character resulting from the presence of zinc ions. These fibres can be used for the manufacture of dressings to be used for wounds in subsequent healing stages. The bacteriostatic effects of zinc can be increased or extended by incorporating typical bactericidal or fungicidal agents into fibres during the fibre spinning stage. We confirmed the efficacy of such a modification in our studies on the preparation of fibres from another fibre-forming polymer [10]. The incorporation of typical fungicidal agents into alginate fibres will be the subject of our further studies.

Copper alginate fibres, in addition to high moisture absorption and bactericidal properties, should show improved electric conduction. These features will make it possible to use copper alginate fibres for compression bandages and various kinds of hospital linen (pads, aprons, caps, masks).

Imparting antibacterial properties to alginate fibres will extend their functionality, and will make it possible to obtain a new generation of dressing materials and textiles designed for various medical applications.

The aim of the present study is to carry out a comparative analysis of the effects of basic fibre spinning parameters on the porous structure, moisture absorption and strength of the fibres from zinc and copper alginates.
The selection of fibre formation conditions aimed at maximising moisture absorption or strength properties will make it possible to prepare fibres from either zinc alginate or copper alginate with optimal properties in respect to the fields of their predicted medical applications.

Table 1. Characteristics of polymer and spinning solution.

Solution concentration	Apparent viscosity dynamic, mPas	Rheological parameters	
		n	k
7% sodium alginate solution	26.19	0.85	24.5

Experimental

Characteristics of spinning solutions

The spinning solutions were prepared from sodium alginate, Protanal LF 60/20 product by FMC Biopolymer AS, with a composition in which guluronic acid radicals predominated in relation to those of mannuric acid. To prepare alginate fibres, 7% aqueous spinning solutions of sodium alginate were used. The intrinsic viscosity determined in 0.1 mol solution NaCl at temperature 25 °C is 4.97 dl/g.

The rheological properties of the spinning solutions were determined by means of a Rheotest RV rotary rheometer. The measurements were carried out within the shearing rate range from 0.2 to 1.3×10^3 s^{-1}, and at shearing stresses from 12 to 3×10^3 N/m^2.

The characteristics of the spinning solution are given in Table 1.

The value determined of the rheological parameter n for the solution under investigation is lower than unity, which confirms that this is a non-Newtonian fluid rarefied by shearing without a flow limit. It is characteristic of sodium alginate solutions that they show high values of apparent dynamic viscosity at quite low concentrations.

Fibre formation

Both type of alginate fibres were spun with the use of a laboratory spinning machine[12] that made it possible to stabilise them technological parameters at a predetermined level and keep them under continuous supervision.

The fibres were spun from solution by the wet process using a spinneret with 500 orifices of 0.08 mm in diameter.

The solidification of zinc alginate fibres was performed in baths containing 3% of zinc chloride, and that of copper alginate fibres in baths containing 3% of copper chloride and 0.3% of hydrochloric acid, at a temperature of 22°C. The fibre drawing process was carried out in two stages; in a plasticising bath containing 3% of zinc chloride or copper chloride at 70°C, and in superheated steam at a temperature of 140°C, which created beneficial conditions for the deformation processes.

The fibres were taken up continuously in the form of a bobbin package. Once the residual solidification bath was rinsed off, the fibres were dried.

Testing methods

The fibre properties were assessed on the basis of the following determinations:

- ***Moisture absorption*** under conditions of 65% and 100% relative humidity was determined in accordance with Polish standard PN-71/P-04635.
- ***Water retention*** was determined by the centrifugal method according to Polish standard PN-85/P-04761/04. Water retention was measured by the centrifuge method. Fibre samples were immersed in distilled water containing a surface-active agent (Rokafenol Nx-3 in an amount of 0.1%) for 24 h, and then the absorbed water was centrifuged off for 10 min at an acceleration of 10,000 m s^{-2}.
- ***Fibre porosity*** was determined with the use of a mercury Carlo-Erba porosimeter linked to a computer system to record the numerical values of the parameters determined.
- ***Fibre strength*** properties were determined with the use of an Instron tensile testing machine with series IX software.
- ***Linear density*** in tex was determined according to Polish standard PN-72/P-04758.
- ***Fibre electric conduction*** was determined in a screen measuring system using flat rigid electrodes. Fibre resistivity was established on the basis of the measured intensity of the conduction current in the electrostatic field with an intensity of 2.0 kV/cm. The conduction current's intensity was determined on the basis of the absorption current corrected by the current of depolarisation (measurement time: 1 min.). AnKeythle type 6100 electrometerand a Stratron type 4218 stabilised power supply,were used in the measurements.
- ***Substitution degrees of zinc and copper ions*** in alginate fibres were determined by the spectrophotometric method using a Spekol 11 spectrophotometer [13].
- ***The antibacterial effects*** of fibres were determined according to standard JIS L 1902:2002.

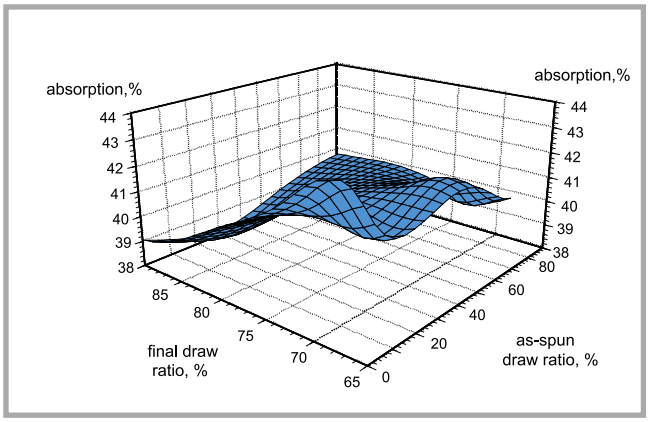

Figure 1. Dependence of the moisture sorption at 100% RH on the as-spun draw ratio and the total draw ratio for copper alginate fibres.

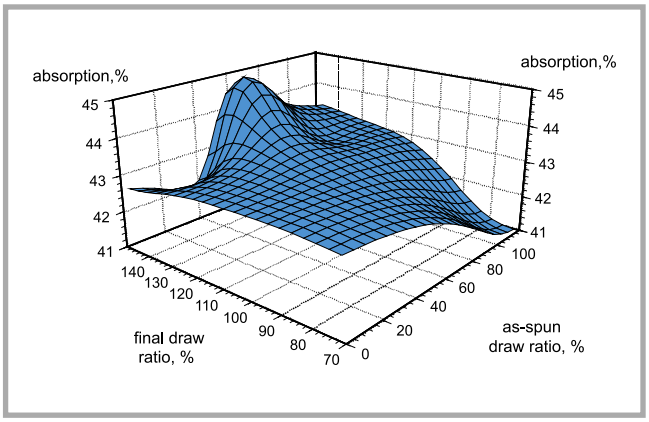

Figure 2. Dependence of the moisture sorption at 100% RH on the as-spun draw ratio and the total draw ratio for zinc alginate fibres.

Results and Discussion

Fibre properties depend on both the chemical composition of the fibre-forming polymer and the structure formed during solidification, as well as the extent of deformation during fibre drawing.

In the case of alginate fibres, the course of solidification depends on the replacement of sodium ions with bivalent metal ions of copper or zinc. The rigid structure of the macromolecule limits the polymer's deformability during fibre drawing. Thus, the orientation of macromolecules taking place in what is still a partly liquid stream is of great importance, as it is dependent on the value of the longitudinal velocity gradient linked directly with the value of the as-spun draw out ratio. The latter parameter, as was previously established [10], usually has contrasting effects on the moisture absorption and strength of fibres.

From the analysis of the effect of the as-spun draw out ratio on the moisture absorption of copper alginate fibres, it follows that this absorption at both 65% and 100% RH tends to decrease with the increase in the as-spun draw out ratio (Figures 1 and 2). The different character of changes in both these indicators versus the parameters under investigation is observed in the case of zinc alginate fibres. The highest values of moisture absorption at 65% and 100% RH are reached by the fibres spun with high values of as-spun draw out ratio above 90%. The moisture absorption at 65% RH of both types of fibres changes within a narrow range from 1 to 2%, assuming higher values (about 18%) for zinc alginate fibres than those for copper alginate fibres (14.5 to 16%). Zinc alginate fibres also show moisture absorption values at 100% RH that are higher by about 7% in comparison with those of copper alginate fibres, the values being 35-39.6% and 32.5-33.8% respectively. Similarly, the water retention of zinc alginate fibres is considerably higher (88.5%) than that of copper alginate fibres, whose retention amounts to a maximum of 63% (Figures 3 and 4).

The character of changes in this indicator versus the parameters examined is similar for both types of fibres, and shows a 3D-dependance (Figures 3 and 4) with a maximum within the range of low values of the as-spun draw out ratio, while a downward trend is observed within the range of the high as-spun draw out ratio. Both types of fibres reach the highest values of retention with the as-spun draw out ratio at a level of 30%. Generally, the changes during the drawing stage in the as-spun draw out ratio, and consequently in the related deformation, affect the moisture absorption of alginate fibres to a considerably lesser degree than those in the case of fibres made from a hydrophobic polymer [10].

It may be assumed that the high values of moisture absorption of alginate fibres, both at 65% and 100% RH, are mainly connected with the hydrophilic nature of their fibre-forming material. With the low total volume of pores (Table 2), in both types of fibres at a level of 0.04 to 0.1 cm^3/g, and an insignificant content of small pores capable of absorbing moisture by capillary condensation, this effect only influences the moisture absorption at 100% RH to a minor extent.

On the other hand, the high values of water retention may be explained as follows.

In accordance with the complex water structure, as reported by many authors [11], water assumes an ordered spatial structure due to numerous interactions resulting in the formation of hydrogen bonds. Water molecules do not adhere closely to each other, and there are considerable free spaces between them due to the shape of the water molecule and the orientation of hydrogen bonds. The gradual attachment of subsequent molecules results in the formation of a polymolecular ordered associate, a so-called cluster. Clusters have a relatively large surface. The hydrogen bonds inside a cluster are more stable than those occurring on its surface. Such clusters are separated from each other by one or two layers of single molecules. They are also capable of decomposing and continuously forming new associates. In the case of strongly hydrophilic alginate fibres, such associates may be attached to the internal surface of the capillary followed by its considerable filling.

However, the character of the porous structure of alginate fibres is characterised by the occurrence of a high maximum comprising the terminal range of large pores and the initial range of very large pores (Figure 7); the latter content predominates and amounts to 60-82%.

In the case of a hydrophobic material, as shown by our studies [10], these pores have too large dimensions to be capable of retaining water after the operation of its mechanical removal. Thus, it may be accepted that the high values of retention are connected with the content of pores of the range under discussion. The value of retention is also affected by the penetration of water molecules into the supermolecular structure of the fibre-forming material.

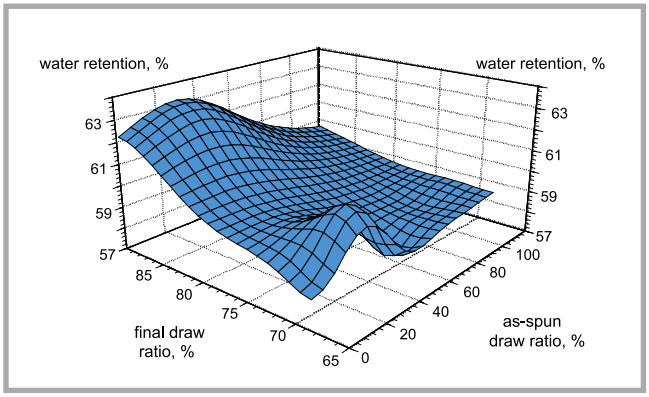

Figure 3. *Dependence of the water retention on the as-spun draw ratio and the total draw ratio for cooper alginate fibres.*

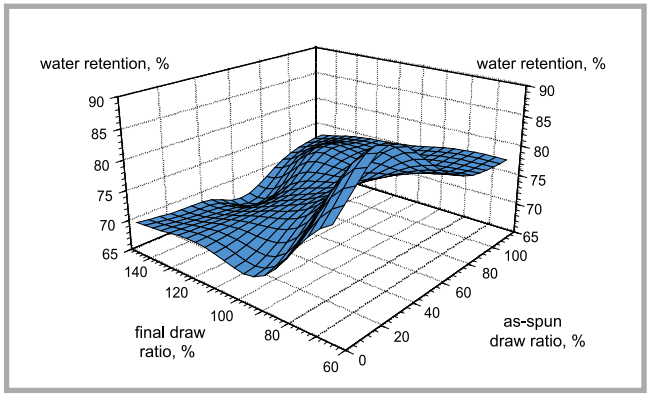

Figure 4. *Dependence of the water retention on the as-spun draw ratio and the total draw ratio for zinc alginate fibres.*

Table 2. *Percentage contents of capillary sets, internal surface and the total pore volume of zinc and copper alginate fibres formed in optimal conditions.*

Fibres from zinc and copper alginate	Total pore volume, cm^3/g	Internal surface of pores, m^2/g	Percentage pore content, %			
			Small 4-12.3 mm	Medium 12.3-75 mm	Large 75-750 mm	Very large 750-7500 mm
Zinc alginate	0.05	1.60	9.76	0.0	7.32	82.92
Copper alginate	0.15	4.98	7.89	14.48	15.79	61.84

Table 3. *Surface resistivity of alginate fibres formed in optimal conditions.*

Type of fibre	Surface resistivity, ρ_S, Ωm/m	
	RH = 25%,T = 23 °C	RH = 65%,T = 25 °C
Zinc alginate fibre	$8.0\times10^{14} \pm 1.3\times10^{14}$	$1.4\times10^{12} \pm 0.2\times10^{12}$
Copper alginate fibre	$3.6\times10^{12} \pm 1.0\times10^{12}$	$1.7\times10^{10} \pm 0.4\times10^{10}$

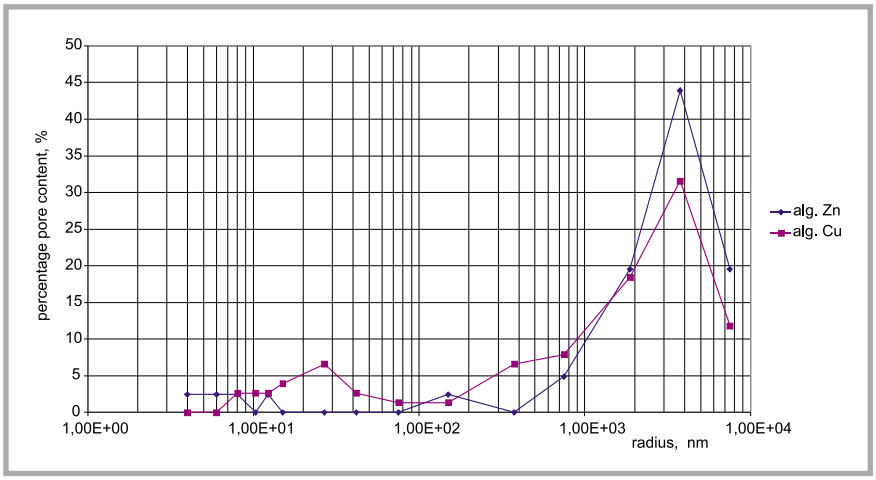

Figure 7. *Curves of pore distribution versus pore radius.*

The combination of adjacent macromolecules with main bonds through copper or zinc brings about the retention of water associates linked to unsubstituted OH groups of the fibre-forming material. This simultaneously limits the typical effect of hydrophilic fibre swelling associated with increased lateral dimensions.

The higher values of retention in the case of zinc alginate fibres can also be partly connected with the higher content (by 20%) of very large pores. However, with the total pore volume being low, it seems that the susceptibility of zinc to form aquacomplexes [14] can affect the relatively high values of retention, as an additional bonding of water around zinc ions linked to the fibre-forming material cannot be excluded.

In both types of fibres, a general trend can be observed towards a increase in strength with the increase in the as-spun draw out ratio (Figures 5 and 6). The highest tenacity of zinc alginate fibres at a level of 28.65 cN/tex was obtained with the as-spun draw out ratio amounting to 90.5%, while the highest tenacity of copper alginate fibres amounting to 21.41 cN/tex was reached at an as-spun draw out ratio equal to 119.9%. The level of tenacity of zinc alginate fibres is higher by about 5-7 cN/tex from that of copper alginate fibres. A similar character of changes in tenacity versus the parameters examined was found in previous studies [12].

Generally, in both types of alginate fibres, the considerable effect of the as-spun draw out ratio and the related deformation during the drawing stage on the fibre tenacity can be observed. The change in the as-spun draw out ratio from 0.1 to 119.9% results in an increase in tenacity by 10 cN/tex for zinc alginate fibres, and by about 5 cN/tex for copper alginate fibres.

The higher tenacity of zinc alginate fibres is connected with the somewhat better susceptibility of the fibre-forming material to deformation during drawing. This makes it possible to obtain a higher total draw ratio with practically the same values of the as-spun draw out ratio.

With similar, low degrees of crystallinity of fibres from copper and zinc alginates, amounting to 9.5% and 5.8% respectively, and crystallite dimensions at a level of 16-18 A, the higher tenacity of zinc alginate fibres is thus associated with better orientation of macromolecules in the amorphous regions of the fibre-forming material.

From the comparative analysis of the surface resistivity of fibres determined un-

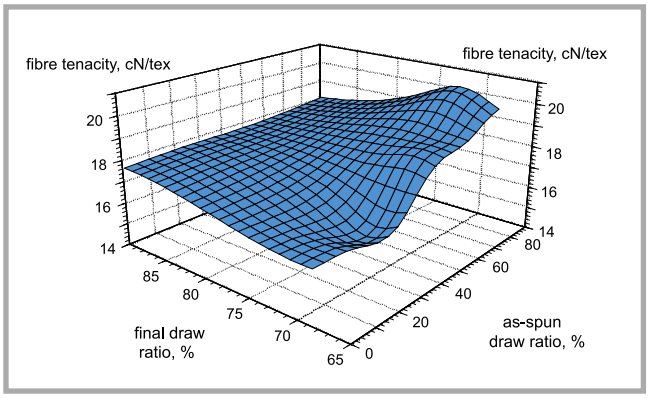

Figure 5. *Dependence of the tenacity on the as-spun draw ratio and the total draw ratio for cooper alginate fibres.*

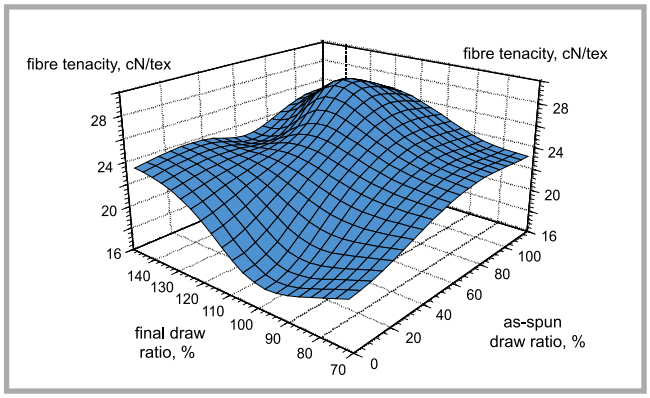

Figure 6. *Dependence of the tenacity on the as-spun draw ratio and the total draw ratio for zinc alginate fibres.*

Table 4. *Zinc and copper ion contents in the fibre-forming material formed in optimal conditions.*

Type of fibres	Ion content in the fibre-forming material, % by wt.
Zinc alginate fibre	8.62
Copper alginate fibre	8.41

der conditions of dry and moist climates (Table 3), it follows that copper alginate fibres show a higher electric conduction than that of zinc alginate fibres. The surface resistivity of copper alginate fibres is lower by two orders of magnitude, both in dry and moist climates, than that of zinc alginate fibres. Therefore, textiles made from copper alginate fibres will show a higher capability to discharge electrostatic charges.

Analysing the content of zinc and copper ions in alginate fibres, we can state that it is at a similar level for both types of fibres, amounting to about 8.4-8.6% by wt. Because the atomic weights of Zn and Cu are similar, the fibre-forming materials of both types of fibres also contain similar quantities of the respective ions. This content is lower than the maximum, that is, at a level of about 15%. This may be associated with the fact that the layer of the substituted polymer with quite a compact structure which appears on the stream surface during solidification hinders further diffusion of the bivalent metal ions. The lowered mobility of the macromolecules combined with the main bonds is also a hindrance to the process of further substitution.

The good antibacterial properties of the alginate fibres under investigation have been confirmed by the assessment of antibacterial effects of textiles on the based on a quantitative test according to standard JIS L 1902:2002, using the bacterial strains Escherichia coli ATCC 11 220 (Gram-negative bacteria). A standard cotton woven fabric was used as reference. The test results (Table 5) have shown that both types of alginate fibres are characterised by high bacteriostatic as well as bactericidal activity.

Practically the same level of bacteriostatic and bactericidal activities of both types of alginate fibres coincide with their similar degree of substitution with Zn and Cu ions at a level of 8.4-8.6%.]] On the other hand, a considerable growth of bacteria was found on the reference cotton fabric.

An estimated indicator of antibacterial characteristic can be valued as a bacterial reduction zone/ground or a number of active bacteria. According to the classification shown in article [15], the antibacterial activity of alginate fibres log(cfu) >3±0.5 can be concedes as being strong.

Based on the examinations performed with the use of a computer-aided experiment, the best conditions for spinning zinc alginate fibres have been selected in respect of the highest values of moisture absorption, as these fibres are designed for a new generation of highly absorptive dressings. The incorporation of zinc into the fibre structure during fibre formation results in its permanent combination, and the resultant fibre shows an antibacterial character.

To obtain maximally high values of water retention (over 88%, of zinc alginate fibres), low positive values of the as-spun draw out ratio at a level of 30% and a decreased value of deformation during drawing must be employed. The tenacity of these fibres, amounting to 21.2 cN/tex, gurarantees their good processing into dressing materials.

In the case of copper alginate fibres, the best fibre spinning conditions have been selected in respect of strength properties. The fibre formation process should be carried out with as high positive values as possible of the as-spun draw out ratio at a level of 120% and deformations close to maximum. The fibres obtained under these conditions show a tenacity above 21.5 cN/tex. At the same time, the fibres show a high absorptive power; their water retention is at a level of 60%, and the moisture absorption at 100% RH amounts to 32.5%. It is assumed that these fibres will be suitable for hospital linen or compression bandages, with additional features such as antibacterial properties and reduced susceptibility to electrostatic charge accumulation.

Table 5. *Results of testing antibacterial properties of alginate fibres formed in optimal conditions.*

Sample symbol	Time, h	Quantity of bacteria, cfu	Confidence interval, cfu	Bacteriostatic activity, log (cfu)	Bactericidal activity, log (cfu)	Growth value, log (cfu)
Standard	0	9.1×10^4	6.7×10^4 - 1.2×10^5	-	-	-
Standard	24	6.5×10^7	5.2×10^7 - 8.8×10^7	-	-	2.8
Zinc alginate	24	47	<1.2×10 - 2.0×10^2	6.1	3.3	-
Copper alginate	24	<20	-	6.5	3.7	-

where: cfu - colony forming units.

Conclusions

- The high absorptive power of alginate fibres with a low total pore volume and an insignificant content of small pores is mainly connected with the hydrophilic character of the fibre-forming material, while the effect of the as-spun draw out ratio and deformation during drawing on absorptive properties is rather limited.
- The decreased susceptibility of the fibre-forming material to deformation during drawing (connected with the rigid macromolecular structure) is a decisive factor in the effect of the as-spun draw out ratio on the strength of alginate fibres. It is beneficial to use high positive values of the as-spun draw out ratio.
- The higher absorptive power, especially the high values of water retention of zinc and cooper alginate fibres, are linked not only to the specific mechanism of water retention within the capillary system, but they also result from zinc and copper's susceptibility to form_aquacomplexes.
- The multi-functionality of alginate fibres consists in combining their high absorptive power with antibacterial and bacteriostatic effects, and in the case of copper alginate fibres, with an improved capability to dissipate electrostatic charges also. The fibre tenacity at a level of 24-26 cN/tex is suitable for the processing into dressing materials and flat textiles for medical applications.
- The selected beneficial spinning conditions for zinc alginate and copper alginate fibres in respect of moisture absorption or strength consist in using decreased values of the as-spun draw out ratio and decreased deformations during the drawing process for the highly absorptive zinc algi-

nate fibres, as well as high values of the as-spun draw out ratio and deformation close to maximum for copper alginate fibres with improved strength properties.

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International Conference
'Fibrous Materials – 21st century'

Saint Petersburg, Russia, 23-27 May 2005

The organiser of this event was the St. Petersburg State University of Technology and Design. The conference was a genuinely broad international forum held in Russia. The Conference program included 74 oral presentations and more than 50 posters; they were presented by speakers from 19 countries. More than 300 participants from 22 countries around the world attended the Conference.

Thanks to the selection carried out by the International Organising and Programming Committee, the advanced subject matter of the presentations created great interest in the participants of this event.

The main topics of the conference were research into fibres, textiles, papers, and composites based on them.

The plenary session included the opening Ceremony and the two following presentations:

- **"The Outlook and Future Developments of Technical Textiles and Man-Made Fibres"**, by *D.E. Morris (Belgium),* and
- **"Development Tendencies in Fibres and Fibrous Materials: Vision of New Technologies and Creation of New Fibre Generations in the 21st Century"**, by *K.E. Perepelkin (Russia)*.

All the remaining presentations were grouped into the following seven blocks:

- Main problems of developing textiles and fibrous composites
- Chemical fibres formation. New fibres.
- Textile technology
- Finishing and dyeing
- Technical and speciality textiles
- Fibrous composites
- Material science: structure, properties and quality control of fibres and textiles

The first block included presentations which focus on important directions in 21st-century research into fibres & textiles.

As an example, some of them are listed below:

- **'Current Trends in Textile Research'**, *H. Planck, E. Singer (Germany).*
- **'Method of Automatic Design of Woven Fabric Definite Constitution Technology'**, *S.D. Nikolaev (Russia).*
- **'Modern State and the Future of Research & Development and Production of Bioactive Fibres for Medical Application'**, *V.A. Zhukovski (Russia).*
- **'Aspects of the Developments of Special Types of Polypropylene Fibres'**, *D. Budzak, M. Jambrich, J. Kochan, M. Revus (Slovakia).*
- **'Production of Cellulose Composites: Present-Day Physical-and-Chemical Issues'**, *L. Akim (Russia).*
- **'Evolutionary Algorithms in Optimal Design of Composite Structures'**, *A. Muc, W. Muc (Poland).*

This was the first time that a conference on present-day fibres and fibrous materials had been organised in Russia, and was accompanied by an exhibition of presentations from different companies and their achievements, and elaborations from universities related to the conference topics. Some interesting discussions between specialists of different countries took place during the Conference. The Conference National Committee also organised some excursions for the conference guests in the city and the surroundings of Saint Petersburg.

The organisers' intention, based on the opinion of participants and speakers, is to organise the next Conference 'Fibrous Materials – 21st century' of a similar character in May 2007, also in Saint Petersburg, Russia, by the Saint. Petersburg State University of Technology and Design.

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