Effects of the Structure of Polymer and Nanosilica Additive on the Sorption and Electric Properties of Various Alginate Fibers

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ABSTRACT: Effects of the structure of polymer and nanosilica additive on the sorption and electric properties of various alginate fibers has been investigated. It has been found that regardless of the differences in the chemical structure and the presence of nanosilica, the examined types of alginate fibers show similar values of moisture absorption at relative humidity up to 85%. An exception is the fiber from sodium alginate. It has been found that the electric conductivity and the accompanying polarization processes of various types of alginate fibers depend on the chemical structure of the fiber-forming polymer. It has been found that the amount of moisture absorbed by the fiber-forming polymer of alginate fibers exerts a strong influence on their electric properties. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 686–694, 2006

Key words: alginate fibers; electric properties; nanoparticles; nanosilica

INTRODUCTION

The development of alginate fibers has been stimulated to a growing extent by its utilization for the production of novel dressing materials and also used in the treatment of not easily healable postburn, varicose, and bed-sore wounds. This is conditioned by the requirements imposed on modern dressings that are adapted to the type and stage of wound healing.[1,2] In addition to basic features such as high moisture absorption and the capability to be partly gelatinized[3] under the influence of wound secretion, the alginate dressings are also capable of supporting the wound healing process because of the specific properties of mannuric acid.[4,5]

The alginate fibers used for the production of most commercial dressing materials consist of calcium alginate or calcium–sodium alginate. The gelatinizing capability of such dressings depends on the contents of guluronic acid (G) and mannuric acid (M) blocks in the fiber-forming material. The character of the resultant hydrogel (rigid or elastic gels), its thermomechanical properties, and dynamic modulus depend on the M/G block ratio.[6] These parameters are also affected

by the degree of sodium alginate to calcium alginate conversion.

High moisture absorption values, being crucial to the use of alginate fibers as dressing materials, are connected mostly with the hydrophilic character of the polymer. On the other hand, the effect of the porous structure created during the fiber-forming process on sorption properties is considerably smaller than that in the case of hydrophobic fibers, because of the low total pore volume and internal surface.[7]

The presence of zinc or copper ions in the alginate polymer extends the range of multifunctional properties of alginate fibers because of their antibacterial activity.[8] Fibers made from zinc or copper alginate can be designed for the production of dressings to be used in the later stage of wound healing as pain-appeasing bandages or hospital linen, as well as clothes for medical staff. In such applications, the fibers should also show suitable electric properties.

As reported in earlier studies,[9,10] there is a relation between the fiber capability to generate a negative charge in contact with human skin and the pain feeling of patients who used bondages or other products, e.g., antirheumatic blankets, with such properties. Based on clinical examinations, it has been found that the generated negative charge of static electricity is a pain appeasement factor in various kinds of pain: neuralgia, post-traumatic, or rheumatic pains. Thus, one may suppose that, also in the case of dressings

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made from fibers capable of generating negative charges, their use will help patients to appease pains.

The electric properties of alginate fibers can be connected with the polymer structure, including alginic acid or sodium alginate, as well as alginate substituted with divalent metal ions during fiber formation. Another factor affecting these properties can be the character of the created porous structure or the quantity of nanosilica incorporated into calcium alginate fibers. Such fibers can be a suitable precursor for the preparation of carbon fibers, designed for implants capable of supporting the process of bone reconstruction, because of the presence of calcium and silicon in their composition.

The aim of the present study is to assess the relationships between the chemical structure of the fiber-forming polymer, its capability to absorb moisture, and electric properties of various types of alginate fibers. The effect of a silica nanoadditive incorporated into calcium alginate fibers on these fiber properties will be also examined. It is the specific resistance that was assumed to be the basic physical feature for the determination of fiber susceptibility to electric charge relaxation.

Considering the high capability of alginate fibers to absorb moisture and the close relation between fiber resistivity and moisture absorption, the examinations will include sorption and desorption isotherms for various types of alginate fibers. This will make it possible to assess the changes in fiber resistivity under various conditions of moisture absorption from a dry to wet condition and desorption ("drying up") under isothermal conditions that could approximately correspond to those existing during a prolonged presence of dressing on a wound. The therapeutic effects of various alginate fibers capable of generating static charges will be also examined.

Object of studies

The object of our studies included alginate fibers of various structures, which are designed for the following medical applications:

- Dressing materials—fibers from alginic acid, sodium alginate, and zinc alginate.
- Hospital linen and pain-appeasing bandages—copper alginate fibers.
- Nanosilica-containing calcium alginate fibers as precursors for carbon fibers designed for implants capable of supporting the process of bone reconstruction due to the presence of calcium and silicon. The quantities of nanosilica incorporated into spinning solutions amounted to 3 and 5% in relation to polymer. The nanoparticles of the silica used had dimensions from 50 to 100 nm. The thermal properties of the fibers from the point of

view of polymer structure and nanoadditive content have been considered in our previous paper[11] that includes also the characteristics of the porous structures and selected properties of particular types of fibers.

- The alginate fibers were spun under conditions that were selected as the most beneficial for fiber applications. The optimization of fiber-spinning conditions was carried out with the use of computer-aided experiment design system, including the maximization of moisture absorption (fibers for dressing applications) or strength properties (with good sorption properties) in the case of fibers designed for hospital linen.
- In the case of precursor fibers, it was intended to obtain good strength properties and increased porosity at the same time, as it is a beneficial with regard to an easier growth of cells on carbon fibers.[12]
- Fibers were spun from 7 to 7.5% sodium alginate (Protanal 20/60) solutions, containing mostly guluronic acid blocks. The intrinsic viscosity of sodium alginate, determined in a 0.1 mol NaCl solution, was 4.97 dL/g. The apparent dynamic viscosity of spinning solutions ranged from 42 to 43.
- The solidification of sodium alginate fibers was carried out in a bath containing ethyl alcohol, under conditions described in our earlier study.[13] The solidification of fibers from alginates substituted with divalent metal ions was performed in a bath containing chlorides of these metals in a quantity of 3%. The solidification of alginic acid fibers took place in a bath containing 2% HCl.
- Calcium, zinc, or copper alginate fibers were drawn in two stages: in a plasticizing bath of the same concentration as that of the solidification bath at 50–75°C and under superheated steam at a temperature of 140°C. Sodium alginate fibers were drawn in 75% ethanol solution at 10–15°C, while the drawing of alginic acid fibers took place in an aqueous plasticizing bath at 50–70°C. After rinsing, the fibers were dried at 20–60°C under isometric conditions (sodium alginate fibers were not rinsed). The characteristics of fibers are given in Table I.

EXPERIMENTAL

Sorption and desorption

The moisture absorption of fibers was measured at various values of the relative air humidity at a temperature of 23°C: 25, 40, 50, 65, 80, and 90%, varying them toward ascending values (sorption) and then toward descending values (desorption).

Fiber porosity was measured by means of a mercury porosimeter of Carlo-Erba linked with z computer

system, to register the numerical values of the measured parameters. The determinations included: the total pore volume, total internal surface, volume of capillary group with a defined radius, and pore percentage content. This method allows one to determine the pore percentage content with the given ranges in the capillary set, with sizes of 5–7500 nm.

Fiber crystallinity was determined by the WAXS method, using a Seifert URD-6 diffractometer, Cu Kα radiation, and a nickel filter. The accelerating voltage was 40 kV and anode current intensity 30 mA. The X-ray diffractograms of fiber samples were taken within the 2θ range from 4° to 60°, with step 0.1° and count time 10 s.

To determine the content of crystalline phase, the diffraction curves were divided into two components of scattering from amorphous and crystalline regions, and the surface areas under the curves (A_a and A_k) were calculated. The content of crystalline phase was calculated from the following equation:

$$X_k = \frac{A_k}{A_k + A_a}$$

The diffractograms were analyzed with the use of Hindeleh and Johnson's[14] method, while its processing was carried out by means of a suitable computer program using Rosenbrock's method.[15] The measurements were performed at the Academy of Technique and Arts in Bielsko Biała.

Fiber tenacity and elongation at break were measured according to the Polish Standard PN-85/P-04761/04, referring the breaking force to the fiber linear density in tex.

Resistivity measurements

A bundle of 500 elementary fibers was placed between flat strip electrodes,[16] and after initial depolarization to an electrically neutral sample, a constant voltage was applied, and the intensity of total current was recorded for 1 min. Then, the electrodes were earthed, and the intensity of depolarization current was recorded for 1 min. The resistivity values of dry (variant a) and wet (variant b) fibers were measured. The fibers were dried at a temperature of 70°C to a constant weight, and then they were conditioned under the atmosphere of predetermined relative humidity (RH) parameters. In variant a, fibers were placed in a vacuum chamber with a vacuum of 0.7 Torr for 10 h, and under these conditions, the total and depolarization currents were measured. Owing to low values of total current, the number of fiber bundles in a sample was increased to 10. In variant b, the fibers were conditioned under isometric condition ($t = 23$°C) at RH: 25, 40, 50, 65, 80, and 98% going toward ascending values

and then toward descending values. The measurements were performed by means of an electrometer, type 610C, of Keithley.

The resistance of fibers was determined from the values of total current ($I_{\text{total 1 min}}$) minus the depolarization current ($I_{\text{depolarization 1 min}}$). Based on the determined values of resistance, the volume resistivity of fibers was calculated from the relationship:

$\varsigma_v = R^*(S/L)$ [Ωm], where R^* is the average resistance of the fiber sample ($R^* = U/(I_{\text{total}} - I_{\text{depol}})$, U the measurement voltage, S the cross-section area of the fiber sample, and L is the distance between the electrodes.

Measurement of static electricity generated by the fibers

The susceptibility of alginate fibers to generate static charges was examined on woven fabrics with a plain weave, which were wrapped around a metal roller fitted with an insulated handle. After neutralization, the roller was rolled on the surface of (a) human skin and (b) artificial skin. After a fixed number of roller-skin contact cycles, the roller was put into Faraday cage connected to an electrostatic voltmeter, to collect the accumulated charges.

The artificial skin used in the measurements consisted of a test material applied as a cover of dummy man to simulate the static charge generation on human body during the use of clothing.[9] (The resistivity of the test material corresponds to that of human skin behavior: $\varsigma_v = 100\ \Omega$m).

RESULTS AND DISCUSSION

Sorption properties of alginate fibers

From the sorption and desorption isotherms shown in Figure 1 for various types of alginate fibers, it follows that their courses are similar in character. Exceptional are the isotherms of sodium alginate fibers, for which, starting from a humidity of 75%, the curve shows a strong upward trend. Also, the desorption process runs differently, which is seen in the narrow hysteresis loop (Fig. 1). Still narrower hysteresis loop is observed in the case of fibers from alginic acid. These differences are associated with the mechanism of bonding and retention of water in alginate fibers of different chemical compositions and internal structures.

A relatively wide hysteresis loop is observed for fibers made from alginates of divalent metal ions Zn, Cu, and Ca. These fibers are characterized by a spatial arrangement of macromolecular fragments of the "eggs-boxes" type.[17] Such a structure makes the water desorption difficult because of its retention in a specifically created structure. It is also possible that
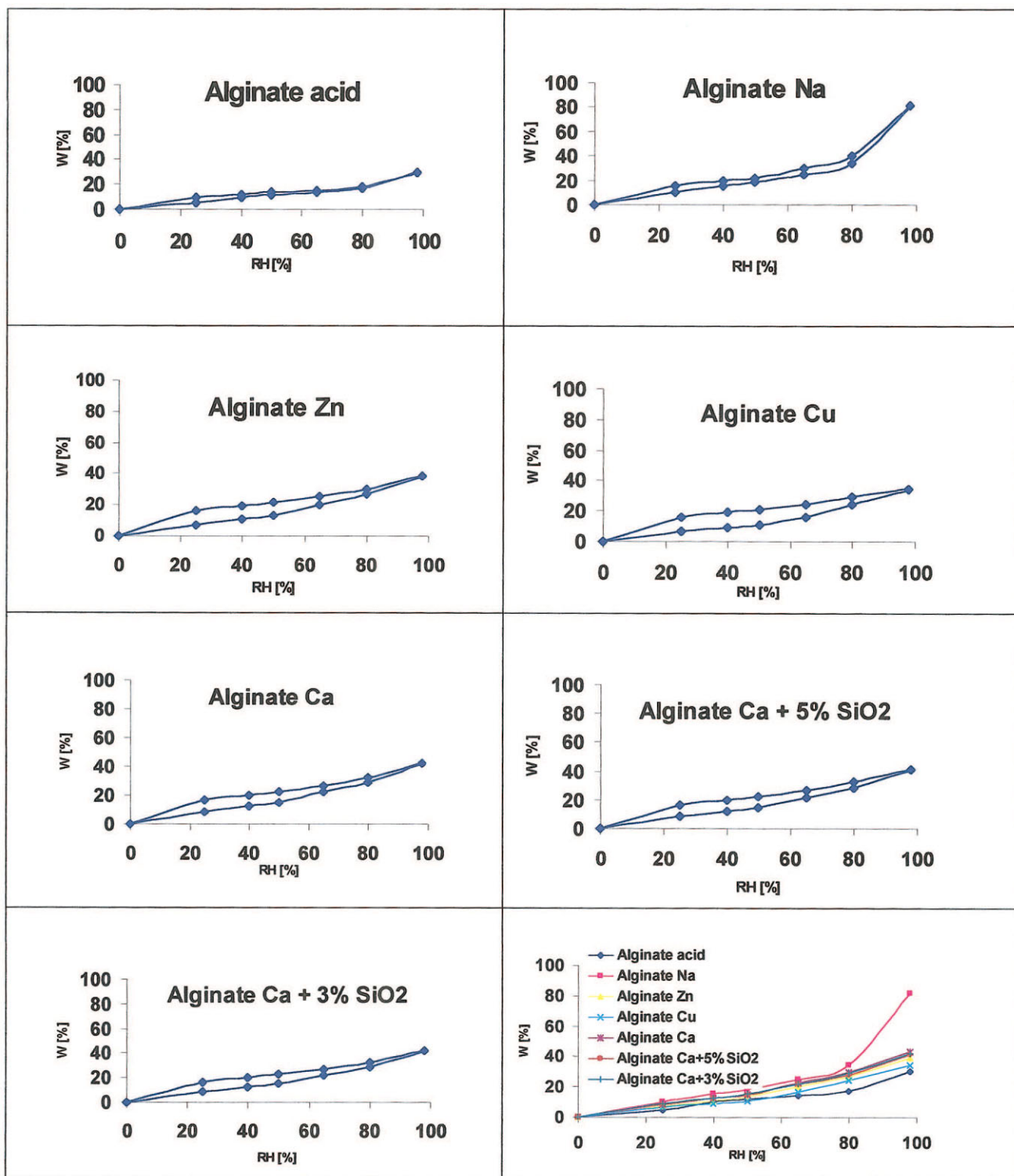
Figure 1 Sorption and desorption isotherms of alginate fibers. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

greater associates are combined into clusters,[18] with unsubstituted OH groups of the polymer. This decelerates the water desorption process during fiber drying.

On the other hand, in fibers made from alginic acid or sodium alginate, whose macromolecules are not combined with each other with chemical bonds, the desorption process runs easily, which is seen in the narrow hysteresis loop. In the case of alginic acid fibers, there is a somewhat greater possibility to combine water through hydrogen bonds because of the presence of COOH groups at C-6.

TABLE I
Structural Parameters and Properties of Various Alginate Fibers Spun under Optimal Conditions

Type of material	As-spun draw ratio (%)	Total draw ratio (%)	The total pore volume (mm^3/g)	Internal surface of pores (m^2/g)	Degree of crystallinity (%)	Tenacity (cN/tex)
Alginate Ca	70.37	54.65	54.32	1.80	8.5	23.53
Alginate Ca $+3\%SiO_2$	60.00	63.66	91.25	4.53	11.2	19.93
Alginate Ca $+5\%SiO_2$	90.23	59.03	90.70	5.53	10.2	19.29
Alginate acid	120.00	17.72	58.73	3.88	27.0	16.05
Alginate Na	5.00	27.00	—	—	11.8	11.55
Alginate Zn	30.50	64.63	47.67	1.60	5.8	21.24
Alginate Cu	119.90	73.23	146.15	4.98	9.5	21.41

In the case of sodium alginate fibers, with higher values of RH the water retention is increased, which results in the dissociation of Na^+ ions, and consequently, in dissolution of these fiber in an aqueous medium. With sufficient amount of retained water, this leads to the conversion of fibers into a gel form. On the other hand, with RH approaching 100%, this phenomenon is shown by a steep increase of the sorption isotherm. Taking into account the low total volume and internal surface of pores in all the types of alginate fibers (Table I), and despite the almost 18% content of small pores with dimensions 4–12.3 nm that are capable of absorbing water through the capillary condensation mechanism, it can be assumed that the water absorbed in the supermolecular structure of the fiber-forming polymer is combined mostly via hydrogen bridges. With comparable degrees of polymer substitution with divalent metals, such as Zn, Cu, and Ca at a level of 8.6–8.4%, the numbers of OH groups in macromolecules capable of creating these bonds are similar, which corresponds to similar sorption properties at a level of 20% at 65% RH.

Slight differences in these values were reflected in the position of isotherms in the following series of ascending values (at 65% RH): Cu, Zn, Ca + SiO_2, Ca (Fig. 1). Similar arrangement is observed for fibers at other RHs used in the measurements. Fibers from sodium alginate are at the end of this series, which is clear in the light of the previously presented interpretation. On the other hand, the lowest value of moisture absorption of fibers from alginic acid and related positions of isothermal curves can be ascribed to the more compact structure created due to the use of higher values of as-spun draw out ratio during fiber formation. The higher value of the longitudinal rate gradient is conducive to the arrangement of structural elements in the liquid stream being under solidification. The fibers from alginic acid obtained under such conditions with a lower value of draw ratio show a lower content of noncrystalline phase as compared to fibers from alginates of divalent metals (Table I).

Small differences between moisture absorption and isotherm positions for fibers from alginates of divalent

metals are consistent with similar values of noncrystalline phases in these fibers (Table I).

Electric conductivity of alginate fibers

The conductivity of various types of alginate fibers in a constant electric field was described on the basis of volume resistivity, determined within the range of proportional current-voltage dependence of the fibers (Table II and Fig. 2).

The results of resistivity indicate a strong dependence of fiber conduction on the climatic condition of the medium. Under vacuum alginate, fibers show features of electro-insulating materials, while in a medium with increased RH they are electroconductive materials. This indicates a considerable effect of the moisture absorbed by the fibers on their electric conductivity.

Under vacuum, no significant differences were found in the values of resistivity of fibers from Zn, Cu, and Ca alginates.

The addition of nanosilica does not change the electro-insulating properties of calcium alginate fibers considerably. The behavior of fibers from sodium alginate and alginic acid in electric field is different from that of the remaining alginate fibers. The conductivity component of total current of these fibers is considerably higher than the polar component, which results in a decrease in resistivity by three orders of magnitude. It may be assumed that in fibers from sodium alginate and alginic acid, the free charge carriers appear in higher concentrations. The number of these free charge carriers increases with increasing amount of water molecules combined with fibers, when they are transferred to a medium with increasing RH (Fig. 2). This results in a violent decrease in the resistivity of fibers. The rate of changes in resistivity for all types of alginate fibers is similar. Sodium alginate fibers and alginic acid fibers show considerably lower values of resistivity within the whole RH range as compared to the fibers from alginates containing divalent metal ions. During fiber drying, there appears a phenomenon of conductivity hysteresis for all types of alginate
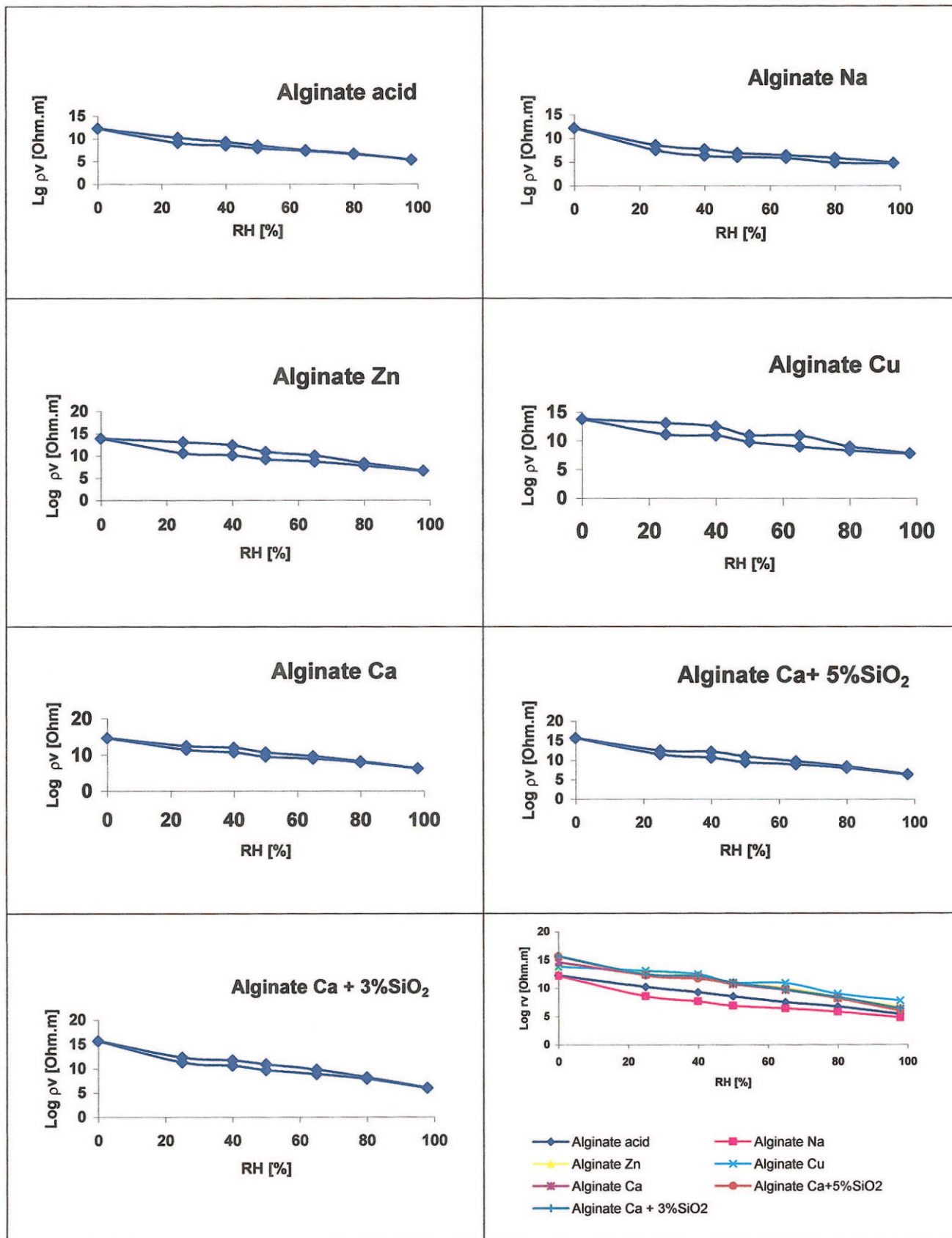
Figure 2 Change in electrical resistivity of alginate fibers with RH. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

fibers. The character of changes in the resistivity of the alginate fibers under investigation is connected with the interaction of water molecules included in the fiber polymer.

The dependence of resistivity of alginate fibers on the moisture content in air and consequently on the content of water molecules in fibers indicates a ionic character of the electric conductivity of alginate fibers. The water molecules combined with a particular alginate are ionogenic, and as the amount of water molecules increases in the polymer, the fiber resistivity decreases. In view of the high content of noncrystalline phase easily accessible to water molecules, in all types of alginate fibers (except alginic acid fibers), the rate of changes in resistivity is almost the same in all of them.

Fibers from alginic acid and sodium alginate show a considerably higher conductivity by three orders of magnitude as compared to that of fibers from Zn, Cu, and Ca alginates. These differences may be due to two reasons: the generation of a higher number of free charge carriers in sodium alginate and alginic acid under the influence of the electric field and their higher mobility. On the basis of the chemical structures of alginic acid and sodium alginate, one may assume that in fibers made from these polymers, hydrogen ions (protons) are the carriers of charges, whose mobility in the electric field is higher than that of OH^- ions ($V^{H^+} = 10^{-8}$ cm^2/s V, $V^{OH^-} = 1.6 \times 10^{-8}$ cm^2/s V). These ions can be generated in the polymer under the influence of the electric field and also under conditions of minimized moisture content (vacuum). In the case of fibers containing water molecules, hydrogen ions can additionally be generated by ionization of hydrogen bonds created by water molecules, with the protons of glutaric acid and mannuric acid radicals. The substitution of proton in alginic acid with divalent metal ions considerably decreases the reserves of free charge carriers, which is seen in the increased conductivity of the fibers.

Polarization of alginate fibers in the electric field

The total current flowing in alginate fibers shows a transient character with time, which indicates the appearance of polarization processes in the fiber-forming polymer. The kinetics and amplitude of changes in the total current are diversified and depend on the type of alginate and the state of physical medium. The susceptibility to polarization was determined on the basis of the intensity of depolarization current recorded in fibers, 1 min after the initial polarization of fibers in the electric field (Table II). The depolarization current intensity measured under vacuum was found to be 2×10^{-14} A for fibers from alginates substituted with divalent metal ions and 2×10^{-13} A for fibers from sodium

TABLE II
Electric Parameters of Various Types of Alginate Fibers[a]

Type of fiber material	Volume resistivity (Ωm)	Depolarization currents (A)
Alginate Ca	8.9×10^8	-2.0×10^{-13}
Alginate Ca +3%SiO$_2$	6.2×10^8	-2.1×10^{-13}
Alginate Ca +5%SiO$_2$	1.8×10^{11}	-0.2×10^{-13}
Alginate acid	1.3×10^{11}	-0.2×10^{-13}
Alginate Na	1.8×10^{11}	-0.2×10^{-13}
Alginate Zn	2.9×10^{11}	-0.2×10^{-13}
Alginate Cu	2.9×10^{11}	-0.2×10^{-13}

[a] Measured under vacuum. $P = 0.7$ Torr; $E = 0.8$ kV/cm.

alginate and alginic acid. On the other hand, no effects of the type of divalent metal ion combined with the polymer and of the presence of nanosilica in calcium alginate on the polarization of fibers were found. The portion of the polarization component in the total current recorded in the fibers is higher after 1 min action of the electric field than the conductivity component. Hence, one can conclude that the combined charge carriers and the spatial charge, on which depends the value of the polar component, dominate in the response of the given alginate to the electric field. A different behavior is shown by the fibers from sodium alginate and alginic acid. The susceptibility of these fibers to polarization is higher than that of alginate fibers substituted with divalent metal ions, with their conductivity also being higher (the processes of conductivity dominate over the polarization processes).

Under moist conditions, a stronger diversification of the susceptibility to polarization of various alginate fibers was observed. The intensity of depolarization current increases in sequence from alginates substituted with divalent metal ions and alginic acid to sodium alginate fibers. The latter fibers show a very high depolarization current at a level of 10^{-9} A, while a considerably lower value of this parameter is observed in alginic acid fibers. The depolarization current of the remaining alginate fibers is low, ranging from 10^{-10} to 10^{-11}A, with slightly higher values being observed for calcium alginate fibers containing nanosilica. The presence of nanosilica in calcium alginate fibers increases the susceptibility of these fibers to polarization in a moist medium over 50% RH. The changes in susceptibility to polarization are shown in Figure 3 as a relative change in the polarization current intensity of fibers from calcium alginate containing nanosilica in relation to that of calcium alginate fibers without this nanoadditive. Such a behavior is probably connected with the arrangement of absorbed water in the porous structure of fibers containing dispersed silica nanoparticles in the volume of calcium alginate. This corresponds to the higher porosity of these fibers (Table I).
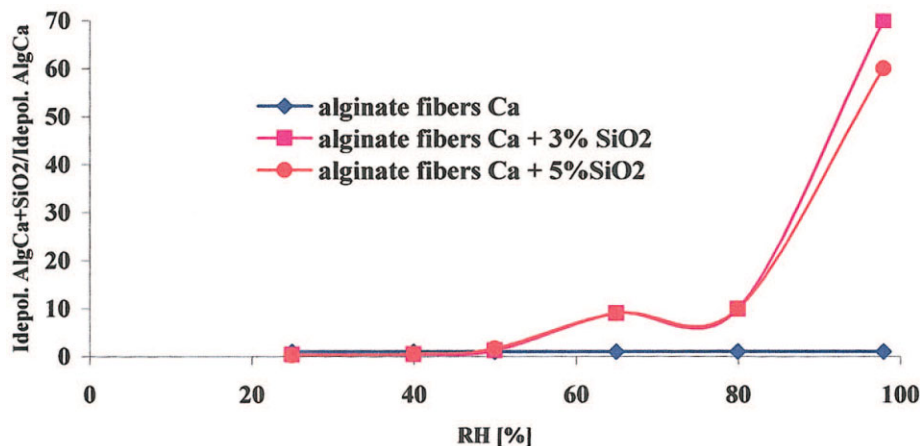
Figure 3 Relative intensity of depolarization current in calcium alginate fibers containing nanosilica. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Chargeability of alginate fibers

Woven fabric samples of various alginate fibers were subjected to static charge generation by contact with human skin and "artificial skin." The procedure consisting in rolling a sample over the skin surface limited the phenomenon of friction, and therefore, reproducible conditions of static electricity were provided. The results of measuring the charges generated in contact with human and artificial skins at 25 and 40% RH and a temperature of 23°C (Table III) are almost similar to each other.

The alginate fabrics in contact with skin assume an excess negative charge. The charge value depends on the raw material composition of the woven fabrics and, to some extent, on the climatic conditions of the medium. Fabrics made from fibers of divalent metal alginates generate charges to a similar extent. The incorporation of nanosilica into calcium alginate does not change its susceptibility to static charge generation, while a clearly higher value of charge is observed in the case of the fabric made from sodium alginate fibers. The density of surface charge generated on fabrics made from alginate fibers was measured in a system that allowed comparison with that of a fabric made from PVC fibers. The therapeutic effects of PVC fibers have been confirmed in numerous studies.[10] According to the authors, the therapeutic properties of PVC fibers are due to the action of the negative charges generated in contact with human skin. Clinic examinations have also confirmed that the negative charge can appease pain. Considering the fact that fabrics made from alginate fibers generate negative charges in contact with skin, one may assume that fabrics made from these fibers will show similar effects to those of PVC fabrics.

CONCLUSIONS

1. Regardless of the differences in the chemical structure and the presence of nanosilica, the examined types of alginate fibers show similar values of moisture absorption at RH up to 85%. An exception is the fiber from sodium alginate that absorbs above that RH value twice as much of moisture as other fibers. With low total pore volume and internal surface, the sorption properties of alginate fibers depend on the hydrophilic character of the fiber-forming polymer. A characteristic phenomenon is

TABLE III
Chargeability of Woven Fabrics Made of Various Alginate Fibers

	Surface density of charge (C/cm^2)			
	Human skins		Artificial skins	
Type of fabric	RH = 25%	RH = 40%	RH = 25%	RH = 40%
Alginate Na 100%	-4.8×10^{-11}	-1.8×10^{-11}	-4.0×10^{-11}	-2.6×10^{-11}
Alginate Zn 100%	-2.4×10^{-11}	-1.0×10^{-11}	-2.4×10^{-11}	-1.0×10^{-11}
Alginate Cu 100%	-2.6×10^{-11}	-0.9×10^{-11}	-2.4×10^{-11}	-1.0×10^{-11}
Alginate Ca 100%	-2.6×10^{-11}	-1.0×10^{-11}	-2.4×10^{-11}	-1.0×10^{-11}
(Alg. Ca + 3% SiO$_2$) 100%	-2.7×10^{-11}	-1.0×10^{-11}	-2.4×10^{-11}	-1.0×10^{-11}
PVC 100%	-15.6×10^{-11}	-14.0×10^{-11}	-15.3×10^{-11}	-14.0×10^{-11}

the appearance of the hysteresis of moisture content of fibers. The substitution of alginates with divalent metal ions results generally in the retardation of water desorption processes.

2. The electric conductivity and accompanying polarization processes of various types of alginate fibers depend on the chemical structure of the fiber-forming polymer. In a medium with minimal humidity (vacuum), the fibers from alginates of divalent metal show a resistivity higher by three orders of magnitude than that of sodium alginate and alginic acid fibers. All the types of alginate fibers under investigation show under such conditions electro-insulating properties.

3. It has been found that the amount of moisture absorbed by the fiber-forming polymer of alginate fibers exerts a strong influence of their electric properties, which change from electro-insulating to electroconductive properties, with the extent of this change being dependent on the air RH.

4. Fibers made from various types of alginates generate negative static charges when being in contact with human skin. The fibers from alginates of bivalent metals and those containing nanosilica show a lower level of static charge than that of charge generated on the fibers made from sodium alginate. The level of the generated charge and its negative polarization can decide about the enhancement of the therapeutic effects of alginate fibers.

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