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Mechanoacoustic Method for Production of Composite Chitosan Finishing Agents for Textile Materials

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Abstract—Theory and practice of fibrous materials modification using chitosan finishing agents with functional additives are discussed. Efficiency of the mechanoacoustic method for production of composite finishing agents is proved.

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Chitosan is a biologically active polysaccharide of natural origin, possessing a complex of practically valuable properties [1]. The interest of specialists working in the field of textile chemistry to chitosan is caused by such properties of this polysaccharide as solubility in water, nontoxicity, good film-forming properties, beneficial effects on the human skin, and ability for reagent-free fixation on natural fibers. Due to these properties it is possible to consider chitosan a promising finishing material for textile products, giving them special properties as a result of finishing.

Kosygin Moscow State Textile University performs research works aimed to create theoretical bases for application of chitosan in the processes of fibrous materials final finishing. In particular, it is found that finishing of fabrics from natural and mixed fibers with chitosan solutions leads to an increase in their resistance to abrasion, tear, and repeated flexing, as well as in the intensity and durability of colors on the material in case of dyeing and printing with reactive dyes [2–4]. There are interesting works dedicated to finishing of fabrics with chitosan derivatives with long hydrocarbon substituents [5] and to manufacturing of chitosan-containing viscose yarn [6].

In other countries chitosan is used for improvement of textile materials in production of elite extra-comfort textiles and medical textiles. Moreover, the required effect is achieved both by introducing the biopolymer into the fiber [7] and by applying it onto the surface of the material [8]. In Russia at present there are no operating technologies for special finishing of textile materials with the use of chitosan. Taking into account the economic situation in the Russian textile industry, now it is hard to count on application of chitosan in production of consumer goods. This polymer can have closer implementation prospects in the context of production of finishing agents containing active substances and making it possible to provide textile materials with special properties (bactericidal, antifungal, curative, selective-sorption, protective etc.). Chitosan itself possesses the above-listed properties; however, incorporation of special functional additives makes it possible to achieve a more evident desired effect. Application of chitosan-containing composite finishing agents will make it possible to manufacture textile products characterized by increased hygienic properties and extra comfort for people with skin problems, as well as products for medical or industrial use. Such targeted use of chitosan makes it possible to put its unique biological and physicochemical properties into practice, thus, justifying the still high costs of the produced goods.

A special case is introduction of insoluble or partially soluble substances into chitosan finishing agents. Such fillers can be represented by partially soluble therapeutic agents, sorbents, sensitizing agents, flavors, and other products in the state of fine dispersion. The filler can act as a controller of the chitosan matrix structurally dependent properties, such as swelling ability, solubility, and sorption capacity. There are published data on the creation of chitosan-based hybrid materials consisting of the organic (chitosan) and inorganic phases. In the majority of cases the inorganic phase is of nanometer size, which, apart from ensuring special properties, eliminates the possibility of sedimentation [9–12]. Attempts to introduce a regular-sized powdered filler into chitosan solutions leads to a problem of compositional instability of molding suspensions and layered heterogeneity of products obtained on the basis of the dry spinning method [13]. As in the majority of cases polymers have a higher density than chitosan solution, the main technological problem of using low-viscosity finishing agents applied by impregnation is related to sedimentation instability. However, in some cases particles of the filler (soot and poorly wettable organic substances) rise to the surface and a layer depleted of the filler is left in the lower part of the suspension. It is evident that layering of suspensions eliminates the possibility to use them as finishing agents. In case of high-viscosity finishing agents applied by spreading, i.e. applied onto the surface of the textile substrate, the main problem is achieving a high level of the filler dispersion and even distribution of the filler in the polymer matrix.

An efficient way to ensure a high degree of homogeneity, as well as sedimentation and aggregate stability of chitosan-based finishing agents is the mechanoacoustic method of their production using the rotary-pulse apparatus [14–16]. Major components of the activating impact on polymer dispersions in the course of their treatment in the rotary-pulse apparatus are mechanical vibrations of a wide range of frequencies, ultrasonic cavitation, and high shear stresses, affecting the liquid material in narrow air gaps (0.1–0.5 mm) between the elements of the rotor and the stator [17]. In textile finishing production technologies such apparatuses showed good results in obtainment of finely dispersed modified starch thickeners and composite sizing materials [18].

The mechanoacoustic impact on the systems, containing the polymer solution and the filler, leads to great changes both in the water-polymer dispersion medium and in the solid phase. Figure 1 demonstrates the main mechanically initiated processes taking place under conditions of the complex effect of cavitation and shear stresses in dispersions based on polysaccharide solutions. The scheme is based on experimental data obtained in the course of studies performed by the Institute of Solution Chemistry, RAS, dedicated to the hydroacoustic effect on the state of dissolved polysaccharides, in particular, chitosan solutions and suspensions on their basis [14–16, 19, 20]. In the course of the research works it was found that intensive mechanical impacts caused a reduction in dynamic viscosity of chitosan dispersions, which was a combined consequence of a whole range of processes, including a decrease in the polymer solution structuring degree, a decrease in sizes of the aggregates, and partial degradation of macrochains, as well as a reduction in the filler particle size and formation of densely packed adsorption polymer layers. The state of the particles surface can also change in the course of mechanical activation due to cavitation erosion of the surface and opening of juvenile (newly formed) surfaces in the particle size reduction process [21]. The structural homogenization of the dispersion medium, the reduction in sizes of the polymer aggregates in combination with the activation of the particle surface lead to an increase in the number of polymer–filler contacts and intensification of interfacial interactions in accordance with the aggregate and molecular theory of polymer adsorption [22]. The most technologically important consequence of the reduction in the particle size and the intensification of the interfacial interactions in suspensions is an increase in their sedimentation and aggregate stability. Mechanoacoustic dispergation makes it possible to obtain finely dispersed finishing agents stable in time on the basis of chitosan solutions even in cases of poorly wettable (fast popping) or fast sedimenting fillers, when it is almost impossible to obtain a stable dispersion using conventional methods. Kinetic curves of sedimentation of microcrystalline cellulose dispersed in chitosan solution are given in Fig. 2 as an example. Kinetic measurements were performed using the spectrophotometric method (automatic mode) based on the clarification rate (in an experiment on the optical density decrease rate at a wavelength of 400 nm) of the upper layer of the suspensions. Mechanically treated suspensions remain stable for several hours and even days, while in the original suspensions it is possible to observe sedimentation of the filler as early as several minutes after the suspension are made.

As shown by the statistical information survey [23], the leading position in practical application of chitosan and its derivatives both worldwide and in our country is taken by the medical sector. Chitosan is used as a carrier for therapeutic agents [24] and a suture material [25], as well as in capsule shells [26] and surgical dressings [27]. Chitosan possesses unique biological

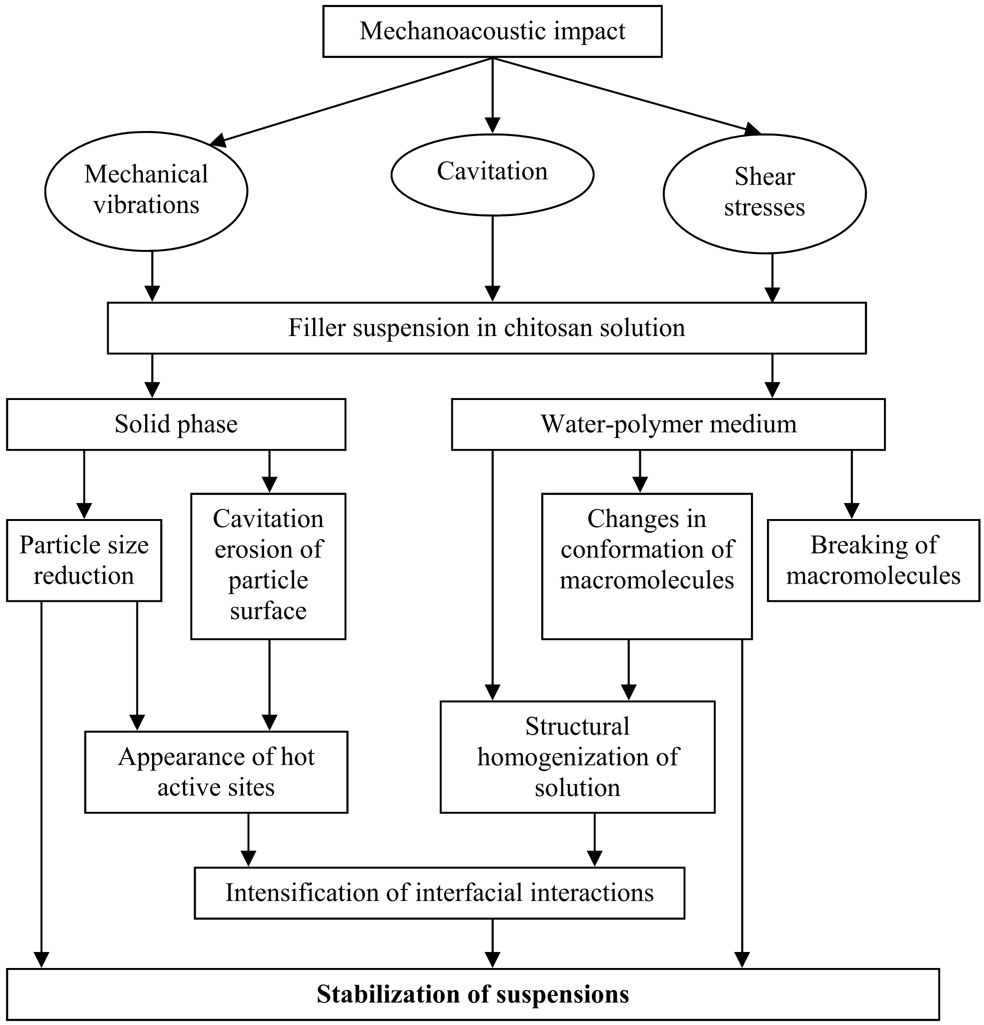

Fig. 1. mechanically initiated processes in suspensions based on polysaccharide (chitosan) solutions under mechanoacoustic impact.

properties, among which it is possible to distinguish its biocompatibility, antimicrobial effect, its ability to activate macrophages, induce cell proliferation for restoration of skin tissues [28] etc. It is known that chitosan-based preparations are used for local treatment of wounds and burns and in wound surgery [29]. Taking into account good thickening and film-forming properties of chitosan, as well as its nontoxicity and high adhesion to natural and certain synthetic fibers, this polymer can be considered the ideal binder-prolongator for fixation of medicines on fibrous substrates. As chitosan possesses its own physiological activity, intensification of activity of the therapeutic agent (synergistic effect) is possible. Soluble medicines distribute well in chitosan solutions. Such gel compositions are applied on porous textile substrates for production of wound coverings [30].

In case of necessity to introduce partially soluble therapeutic agents into the chitosan matrix the required high level of the composition dispersion can be ensured by the mechanoacoustic method of impact. Rotary-pulse apparatuses found application both in textile [18] and pharmaceutical industry [31]; and the use of these devices for production of special finishing agents providing textile products with curative properties can turn out to be highly promising. The possibility and efficiency of applying the mechano-acoustic method of impact for obtainment of chitosan finishing agents containing medicines are described in works [32, 33]. In case of water-soluble therapeutic agents (chloramphenicol, chlorhexidine bigluconate, and furacilin) it is required to perform preliminary treatment of the chitosan solution, which will be later used as a basis for the finishing agent, in the rotary-pulse apparatus.

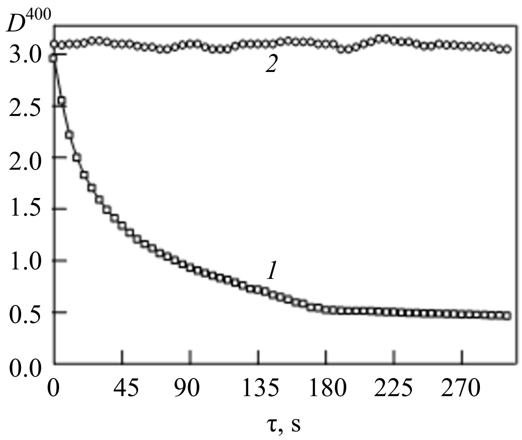

Fig. 2. Kinetic curves of (*1*) sedimentation of original and (*2*) mechanically treated suspensions of microcrystalline cellulose in 2% chitosan solution. Treatment duration 4 s; (D^{400}) optical density of the suspension upper layer at a wavelength of 400 nm.

The objective of the mechanical impact is structural homogenization of the solution and targeted change of the release rate of the therapeutic agent introduced after the treatment from the chitosan film formed on the textile substrate as a result of drying. The changes in transport properties of the polymer film are achieved by variation of parameters of the mechanical impact on the original solution of chitosan. Influence of the treatment duration and intensity, as well as the polymer concentration in the solution on the release rate of the medicine is studied. In case of a partially soluble therapeutic agent (furaginum) its suspension in chitosan solution is subject to mechanoacoustic treatment. In the process disaggregation and size reduction of the therapeutic agent particles take place and even distribution of the filler in the polymer matrix is achieved. Upon contact with the liquid medium extremely small particles of mechanoacoustically incorporated agent slowly dissolve from the surface layer by layer, ensuring great prolongation of the therapeutic effect. Kinetic measurements and creation of histograms on distribution of the therapeutic agent release rate across the area of the treated cloth were used to prove that in case of mechanically activated finishing agents uniform release of the medicine from any site of the material was achieved [32].

Table 1 provides the data characterizing the efficiency of impact of certain medicines in chitosan finishing agents applied to cotton fabrics. The prolongation factor for the therapeutic agent effect was calculated as a ratio of the period of 80% release of the medicine into the aqueous medium from the chitosan film applied onto the fabric to the corresponding period for pure fabric containing the same amount of the therapeutic agent and no chitosan. Indicators of antimicrobial activity of the samples were evaluated according to the bacterial culture growth inhibition zone, which was determined by biological activity and quantity of the added therapeutic agent. Thus, it should be taken into consideration that the therapeutic effect of the medicine can be intensified by an increase in its concentration in finishing agents.

Under conditions of the mechanoacoustic impact it is possible to produce chitosan-containing finishing agents, which cannot be obtained otherwise. First of all, it applies to chitosan–filler systems, in which interfacial complex formation with the participation of chitosan amino groups is possible. It will be demonstrated below with an example of mechanoacoustic incorporation of zinc oxide [34].

Zinc oxide is an antiseptic and fungicidal agent well-known in medicine, which is applied as a binder and drier and included into ointments, baby powders, pastes, hygienic powders for problem feet etc. It is found [35] that chitosan itself possesses distinct fungistatic and weak fungicidal effects and cannot

Table 1. Prolongation effect and biological activity of cotton fabric with chitosan finishing agents containing medicines

Therapeutic agent	Chitosan concentration, wt %	Type of finishing agent	Content of therapeutic agent, wt %		Curative effect prolongation factor		Diameter of growth inhibition zone for bacterial culture (*Staphylococcus aureus*), mm
			liquid medium	on fabric	no activation	after activation	
Chloramphenicol	1.5	Solution	0.085	0.1	7	29	–
Furaginum	4	Suspension	0.2	0.50	–	–	3.6
Furacilin	1.5	Solution	0.2	0.25	9	24	3.9
Chlorhexidine digluconate	1.5	Solution	0.05	0.065	27	80	2.9
Zinc oxide	0.5	Colloidal solution	0.8	1.20	–	–	2.8

compete with well-known fungicides. Zinc oxide is an available and relatively cheap compound; therefore, application of zinc oxide in order to provide textile products with special hygienic and even curative properties can be of practical interest. However, there is a problem related to the fact that zinc oxide is an insoluble powder, which cannot be dispersed in any of the known binders applied in finishing production. Like in solutions of other polymers, zinc oxide forms sticky lumps in chitosan solutions. Depending on the parameters of treatment and the composition of the mixtures under treatment, mechanoacoustic incorporation can result in obtainment of ZnO–chitosan ultrafine dispersions, represented by delicate texture pastes with printing qualities, or stable mesosized colloidal systems. Such dispersions can be applied for finishing of fabrics or nonwoven materials with the use of conventional finishing production equipment.

The specified dispersions include three active forms: nanosized and mesosized particles of ZnO, soluble zinc acetate, and Zn–chitosan complex. The share of each active component is determined by the original composition of the mixture. All these forms display antimicrobial and fungicidal activities. Zinc acetate (antifungal agent) is formed in the system as a result of partial dissolution of ZnO in acetic acid, minor quantities of which are introduced into the mixture to ensure chitosan solubility. In this connection the composition of the mixture also depends on the concentration of acetic acid, determining both the size of ZnO particles (dissolution goes from the surface of each particle) and the ingredients ratio after mechanical activation. High antimicrobial activity of chitosan–zinc complexes with relation to gram-positive and gram-negative bacteria and fungi is confirmed in a number of works [see, e.g. 36]. Apart from that, the efficiency of these complexes as acceptors of oxygen free radicals is also proved [37]. Textile materials (fabrics, nonwoven materials, and gauze cloth) modified with ZnO–chitosan ultradispersed composites are intended for manufacturing of textile products for hygienic and medical use, products with antifungal protection, and extra-comfort goods for people with skin problems.

Application of chitosan from solutions on fabrics and nonwoven materials using the impregnation method can result in an increase in the material rigidity and a decrease in its capillarity and breathability due to the formation of a continuous polymer film on the surface under treatment. It is known that there were certain attempts to obtain a softer and more porous finishing agent using the method of foam application of chitosan derivatives with long-chain substituents possessing increased surface activity [5]. This problem can be solved by making the finishing agent ultradiscrete, as in this case continuity of the finishing agent coating is breached. Discreteness of the finishing agent can be achieved as a result of chitosan transition into the colloidal state.

A possibility to obtain colloidal solutions of chitosan raises great interest among Russian and foreign specialists. Thus, it is reported [38] about the formation of nanosized and mesosized chitosan particles in cavitating media under the impact of ultrasound on chitosan solutions, in which soluble sodium salt with polyvalent anion is additionally introduced. In foreign practice chitosan-containing nanosized particles are used for adsorption of anionic therapeutic agents with the purpose of their controlled release [39].

Stable colloidal finishing agents with nanosized and mesosized chitosan particles are formed as a result of mechanoacoustic impact implemented in the rotary-pulse apparatus in the presence of a precipitation agent [33, 34]. If after the precipitation agent is added the solution is not subject to mechanical activation, the polymer is precipitated in the form of bulky flakes, filaments, or gel. If the process is carried out in the rotary-pulse apparatus, it is possible to achieve the size of colloidal chitosan-containing particles of about 60–300 nm, depending on the concentration and molecular weight of chitosan. Colloidal solutions of chitosan-containing particles obtained on the basis of the mechanoacoustic method were used for treatment of cellulosic substrates, which were put into the colloidal solution for 10 minutes. The surface of the majority of fibrous materials acquires a negative charge in water. Surface ξ-potential of cellulose fibers in water lies within a range of –5 to –40 mV [40]. Therefore, positively charged chitosan-containing particles (ξ = 30–48 mV) are efficiently taken by the fabric surface from the colloidal solution under the influence of electrostatic forces. The micrograph in Fig. 3 illustrates precipitation of chitosan particles on the surface of cotton fiber.

It is found that colloidized finishing agents provide the fabric with ability for strong adsorption of organic molecules, mostly anionic. As model organic substances with large organic molecules the authors of work [32] used C.I. Reactive Blue 21 acid dye and furaginum, a poorly soluble antimicrobial agent. The

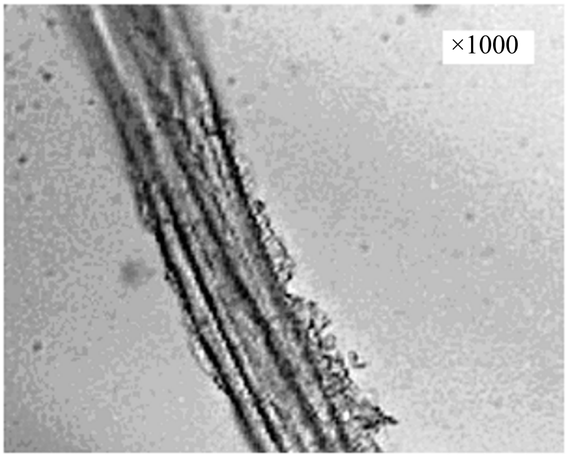

Fig. 3. Micrograph of cotton fiber with immobilized colloidal particles of chitosan 300–400 nm in size.

data given in Table 2 demonstrate that treatment with the colloidal chitosan solution increases the fibrous material ability to adsorb the acid dye by a factor of 20–30, while in case of the true chitosan solution it is increased by a factor of 8–10. The higher effect in case of adsorption of the dye as compared to furaginum can be explained by the fact that in case of ionization in aqueous solutions the dye molecules acquire a negative charge, which contributes to an increase in adsorption on the treated material. As for adsorption of furaginum, electrostatic attraction of its molecules to the treated substrate can be caused by the presence of fractional charges on its non-dissociating oxygen-containing groups. The obtained data demonstrate that the technique of ultra-discrete finishing makes it possible to provide fibrous materials with ability for efficient adsorption of poorly soluble agents from low-concentration aqueous solutions, as a result of which it is possible to achieve a relatively high content of these agents on the textile substrate, at the same time preserving its softness and breathability.

Another important area of chitosan practical application is the production of sorbents. Due to nontoxicity of chitosan the development of chitosan-based sorbents for application in medical practice is very promising. Due to the presence of primary amino groups chitosan forms stable chelate complexes with ions of heavy metals; therefore, it is applied for removal of them from water, drinks, biological fluids etc. [41, 42]. In case of application of granulated and coarse-grain sorbents a long contact with the liquid under purification is required due to unsatisfactory kinetic characteristics of such sorbents, which cannot ensure the required degree of purification at high filtration rates. Powdered sorbents are characterized by better sorption kinetics; however, they create significant hydrodynamic resistance. One of the solutions to the problem of combining a high sorption rate with good hydrodynamic properties of the sorbent is application of the sorbent onto a carrier with a large specific surface area, in particular, onto fibrous materials. There are works on application of chitosan sorbing finishing agents on viscose yarn [6] and wool fiber [43]. Obtainment of chitosan-finished fibrous sorbents is at the laboratory research stage as yet. The mechanoacoustic method can be used for production of composite chitosan-mineral sorbing finishing agents, which after application onto the fibrous carrier ensure a higher rate of sorption of metal ions as compared to purely chitosan agents.

Introduction of certain ultradispersed mineral fillers into chitosan makes it possible to control the polymer matrix sorption properties [9, 22, 44]. However, powdered ultradispersed fillers in chitosan solutions are prone to aggregation; and obtainment of the ultradispersed inorganic phase in these solutions using chemical means [9] involves the formation of associated chemical impurities. Application of chitosan, containing the mechanoacoustically incorporated inorganic filler, onto the fibrous carrier is of great practical interest. In work [45] the fillers used for obtainment of chitosan sorbing finishing agents were represented by barium sulfate, titanium and aluminum oxides. Such choice is related to the ability of small additions of these fillers to reduce viscosity and

Table 2. Adsorption capacity of flax nonwoven material treated with chitosan solutions

Adsorbate and its concentration in solution	Finishing agent	Weight gain based on finishing agent, %	Adsorption value, mg g^{-1}
Anionic dye, 0.004 wt %	– (pure fabric)	–	0.12
	0.1% true solution of chitosan	0.87	1.14
	0.1% colloidal solution of chitosan	0.55	3.50
Furaginum, 0.2 wt %	– (pure fabric)	–	0.12
	0.1% colloidal solution of chitosan	0.45	0.84

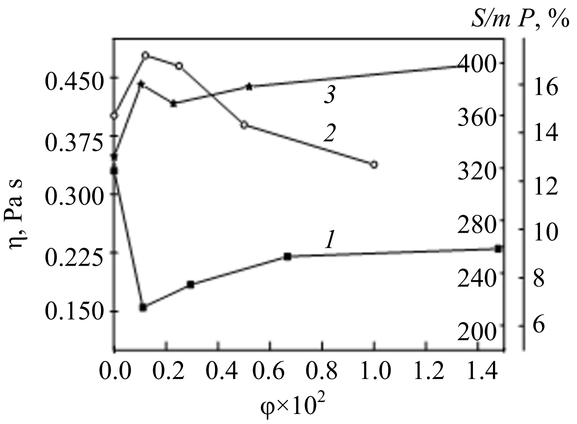

Fig. 4. (*1*) Dependence of viscosity η and (*2*) spreadability *S*/*m* of barium sulfate suspension in chitosan solution and (*3*) dry weight gain *P* in case of suspension Application onto flax fiber from $BaSO_4$ volume fraction.

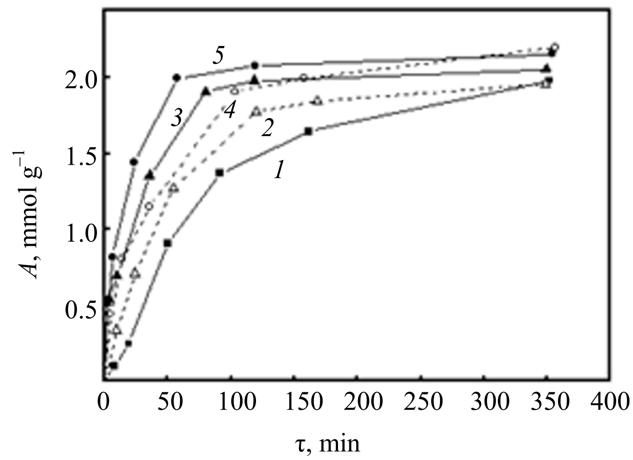

Fig. 5. Kinetic curves of Cu^{2+} sorption by chitosan-containing sorbing finishing agents: (*1*) pure chitosan film (for comparison); (*2*, *4*) finishing agent with no filler on flax nonwoven material (*2*) and on flax fiber (*4*); (*3*, *5*) finishing agent with a filler (5% $BaSO_4$) on flax nonwoven material (*3*) and on flax fiber (*5*).

surface tension of chitosan solutions [46], as well as to increase sorption capacity of films formed from these solutions [15]. The selected fillers have high density, which results in a high rate of sedimentation of particles in inactivated suspensions. Without mechanical treatment of composite finishing agents in rotary-pulse apparatuses, ensuring homogeneity and sedimentation stability, they cannot be used for application to fibrous materials.

Chitosan is a crystallizing polymer; however, it can be expected that under fast drying conditions at high temperatures a great part of the polymer in thin films on the fibers will be in the amorphous state. Introduction of fillers causes a reduction in packing density of macromolecules in the chitosan matrix of composite films, which is known to be a consequence of the limited mobility of polymer chains near the surface of the particles [22]. The reduction in packing density of macromolecules is reflected on chitosan sorption capacity, as there is an increase in the accessibility of functional groups and in the rate of diffusion of the sorbate into the depth of the material. Apart from the reduction in packing density of chitosan macromolecules, a significant role is played by the defectiveness and microscopic piling of the film formed after drying, which also leads to an increase in the adsorbing surface area and, correspondingly, the sorption rate.

Application of chitosan sorbents onto fibrous carriers using the impregnation method is carried out from aqueous solutions. Application of polymer finishing agents onto fibrous materials can result in adhesion of fibers, which leads to incomplete utilization of their surface. In this case the efficiency of the fibrous material impregnation is largely determined by rheological properties and surface tension of the impregnating liquids. It is known that the specific features of the rheological behavior of suspensions, in which the dispersion medium is formed by polymer solutions, are caused by the formation of macromolecules around the particles of the adsorption layer, which leads to an increase in the effective volume fraction of the filler [22]. In case of chitosan, which is a polycation, the character of adsorption interactions in such suspensions is influenced by the particle surface charge. For suspensions with fillers, the particles of which are positively charged ($BaSO_4$, Al_2O_3, and TiO_2), complex dependences of viscosity from the content of the solid phase are obtained [46].

For determination of the optimal concentration of the filler, ensuring the best technological properties of the finishing compound, dependences of spreadability, viscosity, and the quantity of chitosan applied onto the fiber (dry weight gain in terms of the finishing agent on conversion to chitosan) from the content of the filler ($BaSO_4$) in chitosan solution were constructed (Fig. 4). The spreadability indicator *S*/*m*, numerically equal to the ratio of the contact area of the liquid droplet with cellophane film *S* to the droplet mass *m*, in our case characterizes the suspension ability for uniform encapsulation of cellulose fibers. In case of small additions of the filler ($BaSO_4$) it is possible to observe a reduction in viscosity, which after achieving a certain minimal value starts growing again with an increase in the filler content. The plasticizing effect of small additions of the filler in this case can be explained by the fact that solid particles penetrate between supramolecular structures, move them apart, and break a part of bonds between them. Electrostatic repulsion between similar charges of the surface of the filler and the polymer particles facilitates mutual displacement of the suspension structure elements and, correspondingly, reduces the suspension viscosity. Also, the antibonding effect of such fillers on the structure of chitosan solutions manifests itself in a reduction of the suspension surface tension and an increase in the suspension adhesion to the fiber as compared to chitosan solutions containing no filler. The identified effect has a positive impact on the ability of suspensions to penetrate deep into the fibrous material when they are applied by impregnation, ensuring a large specific surface area of the sorbent after drying.

All dependences, presented in Fig. 4, are characterized by the presence of extremum points at a low content of the filler in the suspension, corresponding to 5% content of the filler in dry film. As demonstrated by the production practice of textile materials finishing, the quantity of the applied finishing agent grows with an increase in viscosity of the finishing solution. In our case the maximum weight gain in terms of the finishing agent is achieved at the minimal viscosity of the finishing suspension and the maximal spreadability indicator. It testifies to the fact that under such conditions the maximum weight gain of the fiber is achieved not due to an increase in thickness of the finishing agent film but due to an increase in the total coating surface area, i.e. due to more uniform encapsulation of fibers by the finishing agent and a reduction in their adhesion.

The efficiency of composite chitosan-mineral sorbing finishing agents was evaluated in an experiment studying the sorption of copper ions from aqueous 0.025 M solutions of $CuSO_4$. Figure 5 shows kinetic curves of copper ions sorption by flax fibrous materials treated with chitosan and by chitosan containing 5 wt % of mechanoacoustically incorporated filler $BaSO_4$. A kinetic curve of copper ions sorption on a chitosan film 50 μm thick is given for comparison. As can be seen, the lowest sorption rate is achieved for the chitosan film, while application of chitosan onto the fibrous material increases the sorption rate. Introduction of the filler into the chitosan finishing agent leads to an even higher increase in the sorption rate and the equilibrium sorption value. The period required to achieve the equilibrium sorption is reduced with transition from the purely chitosan finishing agent to the chitosan-mineral composite, as well as with replacement of the nonwoven fabric used as a carrier with fluff fiber.

Thus, it is possible to conclude on the basis of the performed works that the use of the mechanoacoustic impact, which can be implemented in industrial conditions by introducing rotary-pulse apparatuses into the existing processing lines, opens up new possibilities for the production of chitosan-based composite finishing agents, making it possible to provide fibrous materials with unique properties.

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