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MODIFICATION OF FIBRE MATERIALS PROPERTIES WITH THE USE OF NANOTECHNOLOGIES

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Abstract. *Nanotechnologies play an important role in the modern world economy. As they deal, as a rule, together with other convergent (nano-, bio-, info-, cognitive) technologies. This connection causes a synergy effect, i.e. non-linear development of innovations. The growth dynamics of the world's nanotechnology products is impressive. The world market of nanotechnologies in 2021 was 85 billion dollars, in 2024 (plan) will be 140 billion dollars. The forecast for 2030 is $288 billion. The production of nanoparticles of different nature and their use in different industries, fields of science, and technology takes a special place in nanotechnology. Both nanotechnology itself and the production and application of nanoparticles are interdisciplinary and cross-sectoral ones. Their users and customers are developed industries, including the textile industry. Metal nanoparticles are used both in the form of colloidal solutions and as part of microcapsules containing functional substances of different nature in the core. The article considers some methods of obtaining metal nanoparticles and microcapsule synthesis. The authors list the technologies of microcapsules application for textile functionalization.*

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The global textile industry has been one of the main objects of its practical application since the first years (the beginning of the 21st century) when nanotechnology appeared in the real economy. Here is a very vivid statistic of the world nanotextiles production (a broad term - textiles produced with the use of nanotechnology) in US dollars. According to the figures above, the use of nanotechnology in the production of textiles in all its forms is on the top, especially, for technical and military textiles. Indeed, these textiles have seriously increased the demands for both volumes and materials with new properties which nanotechnology can provide. Global production and consumption of all textiles in 2018 is $ USD 780 bn.

Countries major textile exporters in 2018, $ USD bn [1]:

China - 257.8;
India - 37.2;
Bangladesh - 36.8;
Germany - 35.8;
Italy - 35.1;
Vietnam - 34;
Turkey - 26.8;
USA - 25.8;
Hong Kong - 21.3;
Spain - 18.6;
France - 15.7;
Belgium - 15;
South Korea - 13;
Pakistan - 13;
Russia - no data.

Nanotechnology and textiles

Global production of all textiles by nanotechnology [2]:
2016 - $ USD 77.3 bn, 2022 - $ USD 295 bn
Global fabric production by nanotechnology:
2016 - $ USD 50.5 bn, 2022 - $ USD 101 bn.
Global home textile production by nanotechnology:
2016 - $ USD 6 bn, 2022 - $ USD 36 bn.
Global military textile production by nanotechnology:
2016 - $ USD 390 mln, 2022 - $ USD 1.6 bn.
Global medical textile production by nanotechnology:
2016 - $ USD 40 mln, 2022 - $ USD 1.2 bn.
Global sport textile production by nanotechnology:
2016 - $ USD 85 mln, 2022 - $ USD 170 mln,
Global technical textile production by nanotechnology:
2016 - $ USD 20 bn, 2022 - $ USD 155 bn.
Percentage of global textiles produced by nanotechnology:
Fabrics - 10.73%;
Home textiles - 36.59%;
Military textiles - 29.29%
Medical textiles - 21.82%;
Sport textiles - 13.5%;
Technical textiles - 45.1%.

Nanotechnology and textiles

The implementation of nanotechnology for textile production includes [3, 4]:
– production of nanoparticles to provide textiles with special properties;
– production of special purpose fibres using nanotechnology and nanoparticles;
– use of nanotechnology and nanoparticles to provide textiles with special properties;
– developing nano- and micro-containers for the textile materials functionalization.

Production of nanoparticles and their use for special textile properties

The global nanoparticle production of different kinds of nanoparticles in terms of value was $ USD 2.4 bn in 2021, the plan for 2030 is $ USD 6.4 bn. Nanoparticles are nanoscale objects (1-100 nm) possessing a complex of valuable properties, which are determined by their size and shape. Nanoparticles differ in their physical, chemical, physicochemical, biological properties from materials of the same chemical structure in micro- and macro-forms.

New properties of nanoparticles are associated with their small size, which affects nanomaterial surface, its mechanical strength, the ability of nanoparticles to overcome physiological barriers, exhibit unique biocidal, optical-colouristic, electrical, magnetic, quantum, catalytic properties. All the properties nanoparticles possess, they relay, to a greater or lesser extent, to the substrates into which they are volumetrically or superficially incorporated [2].

Methods of nanoparticle production

Nanoparticles appear naturally and artificially; there are two main schemes of their appearing:

– "top-down" by crushing massive material using various physical methods (vacuum, laser, sublimation, etc.);

– "down-up" by associating atoms or molecules into nanoscale associations.

The second scheme is characteristic of the formation of various nanostructures in nature and is close to the phenomena of colloidal chemistry. It is often implemented in man-made nature-like nanotechnologies [5].

Production and use of noble and heavy metal nanoparticles in textile production

Nanoparticles of noble (Ag, Au, Pt) and heavy metals (Fe, Cu, Zn, Ti, etc.) play a special role among all kinds of nanoparticles.

Nanoparticles of these metals show very high biocidal (antimicrobial, antiviral) properties of a wide range action additionally to the positive properties of all nanoparticles. Also, they show quantum effects - acquire colour in a wide range of spectrum depending on the size and shape of particles.

Nanoparticles of noble and heavy metals as well as nanoparticles of other types can be produced by chemical, physical expensive and complex methods, as well as by "green" - environmentally friendly methods (Fig. 1).

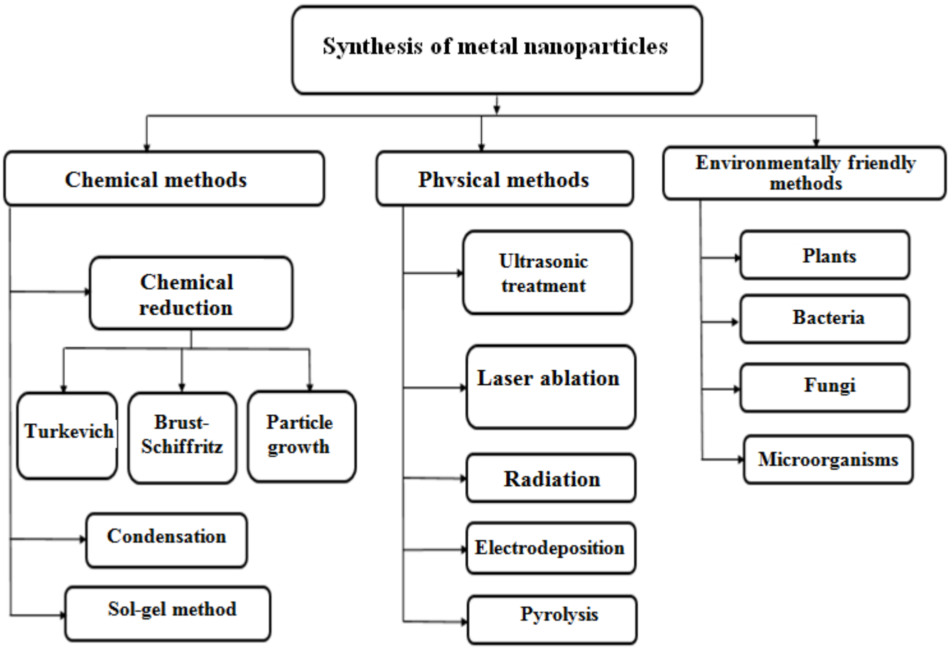

Fig. 1. Classification of synthesis methods for metal nanoparticles [6]

Silver nanoparticles can be obtained by reduction from aqueous solutions of silver nitrate under the action of sodium oxalate, thiourea dioxide, sodium dithionite, oxalic acid dialdehyde (glyoxal), or natural compounds - glucose, ascorbic acid, tannins [7].

The colloidal solutions of nanosilver are proposed to be stabilised by solutions of polyelectrolytes [8]: polydiallyldimethylammonium chloride, polyguanidine, acrylic copolymers, sodium alginate, etc. The minimum size of the synthesised silver particles is typical for glyoxal-polyguanidine systems and is 2-4 nm. Antibacterial properties of textile materials treated with such silver hydrosols, determined by the disc method, are characterised by a zone of bacterial growth inhibition, which is 14-19 mm with respect to *Escherichia coli, Staphylococcus aureus*, 3-7 mm with respect to *Candida albicans* [9]. The proposed formulations are intended for the treatment of medical linen as well as protective masks.

An original, environmentally friendly, nature-based biosynthesis using biopolymers, polysaccharides, has been used to create bandages [10].

Polysaccharides due to their chemical structure (presence of a large number of functional hydroxyl, semi-acetal groups) are able to reduce cations of metal salts (precursors) to the zero-valent atomic state, which further associate to nanoscale formations of metal nanoparticles:

$$MeA \rightleftarrows Me^{n+} + A^{n-}$$
$$Me^{n+} \xrightarrow{reduction} Me^{0\cdot} \overset{0}{\rightarrow} [nMe^{0}] \rightarrow NP\text{Me}$$

The biopolymer (polysaccharide) in hydrogel form fulfils three functions simultaneously:

1. Bioreactor is a platform where the reduction reaction of metal cations to atomic neutral state, association of atoms to nanoscale formations (metal nanoparticles) takes place.
2. A bioreductant possessing functional groups is capable to redox reactions. Bioreductants reduce cations to neutral atoms by oxidising themselves.
3. Colloidal stabilisers of the formed nanodispersion.

Both polysaccharides and proteins of natural fibres can act as bioreductors of metal cations. In this case, metal nanoparticles are formed directly on textiles made of natural cellulose and protein fibres.

Metal nanoparticles obtained by biosynthesis can be used for the production of medical and antimicrobial textiles, etc.: water and air purification, organic catalysis, agriculture, new-generation packaging materials, and modern optics.

Using nanotechnology and nanoparticles in the production of a new fiber generation with special properties [11].

***Production of nanoscale diameter fibres*.**

This technology is based on the principle of jet dispersion (splitting) of a viscous solution or melt of a fibre-forming polymer under pressure passing through a constant electric field. The resulting polymer jets are formed into nanofibres. Indeed, this method (electrospinning/electroforming) can produce nanofibres from all fibre-forming natural [12] and synthetic polymers [13]. The most important issue is to convert these polymers into a viscous solution or melt.

A number of firms produce machines for production of different chemical nature nanofibres [14].

Nanofibres are used as a platform in regenerative medicine in the form of nonwoven materials, in wound-healing applications [15, 16], in the production of filtration (liquid, gas) materials [17]. Such materials are particularly effective in purification of liquid and air from pathogenic harmful microorganisms and viruses.

Filling in the production of chemical fibres with nanoparticles to provide them with special properties [18].

If nanoparticles of different nature and with different properties are introduced into viscous solutions or melts before their passage through the dies of spinning machines, the nanoparticles will transfer their properties to the nanofilledfibre. A composite fibre will be formed with nanoparticles as the main substance of the fibre-forming polymer and filler. This is a volumetric distribution of nanoparticles in the fibre.

Thus, fibres can be provided with increased strength, biocidal [19], magnetic, electrical, photoactive, superhydrophobic [20], flame retardant [21, 22], and other properties. In this fibre production technology, there are problems in passing the composition through the dies of the spinning machine.

It is possible to implement nanoparticle treatment at a later stage of chemical fibre production, namely at the oiling stage, by introducing nanoparticles into the composition of the oiling agent. In this case, the nanoparticles will be deposited on the fibres surface, and it will be necessary to ensure their high adhesion to the fibres for a permanent effect.

The use of nanotechnology and nanoparticles directly in the finishing industry to provide textiles with special properties.

Nanotechnology and nanoparticles are widely used in textile production. It occurs mainly in the final finishing stage (application).

The main properties given to textiles by nanotechnology and nanoparticles in the final finish are: antimicrobial, UV protection, flame retardancy, water repellency, dirt repellency, self-cleaning, healing properties.

The antimicrobial finish is mainly provided by silver and copper nanoparticles using different nanotechnologies (ready-made nanoparticles are applied to the textile and fixed, nanoparticles are synthesised directly on the textile).

– Protection against UV degradation using titanium dioxide and zinc oxide nanoparticles with high photoactivity [23].

– Adding softness to textiles using nanoemulsions [24].

– Water repellent and oil repellent properties using nanohydrophobisers and nano oil repellents [25].

Microencapsulation of functional substances for textile finishing.

Design of nano- and micro-containers for encapsulation of functional substances is a significant trend in the science-based nanotechnology. Microencapsulation is the process of encapsulating a functional substance in a shell protecting it from evaporation, contamination and other environmental influences. It allows the substance to be released in a prolonged manner [26, 27]. The microencapsulation of textile auxiliaries is special reaction.

There are various methods of microencapsulation [28-33]: physical [34, 35], chemical [36-39], and physicochemical [40, 41] ones.

Spray drying is an example of physical methods used for essential oils microencapsulation [42]. Acacia gum, chitosan, etc. are used to form the architecture of the capsule shell. [43]. A common method of encapsulating hydrophobic drugs into water-soluble polymers is the emulsification and solvent removal method [44-48].

Physicochemical methods include simple [49-50] and complex coacervation [51, 52], as well as the molecular inclusion method [53]. The shell-forming polymers in these methods are starch, chitosan, acacia gum, gelatin, and gummiarabic. The core material is oils and colouring agents. β-cyclodextrin is most commonly used for molecular inclusion.

Chemical methods for producing microcapsules consist of synthesising the architecture of the capsule shell around the core by polymerisation or polycondensation of shell-forming substances.

The *in situ*polymerisation process was one of the first proposed chemical methods for the synthesis of microcapsules. It is also actual nowadays [54] and includes emulsion, suspension, precipitation or dispersion polymerisation, and interfacial polycondensation [39]. In this case, direct polymerisation occurs on the surface of a solid particle or droplet; it proceeds at the liquid-liquid, solid-liquid, liquid-gas, or solid-gas interface with the formation of a capsule shell architecture.

*In situ*polymerisation occurs place in oil-in-water emulsions, which allows the formation of microcapsules with a hydrophobic core immiscible with water. It results in spherical, reservoir-shaped microcapsules with smooth, transparent, strong, and pressure-sensitive shells. This method is the simplest one. The formation of emulsions can be accelerated by various physical effects, such as ultrasound [55].

In 1959, the interfacial polymerisation method was first discovered as an alternative to the high-temperature, low-pressure melt polymerisation technique. During microencapsulation by interfacial polymerisation, one of the monomers is dissolved in an aqueous phase and the other in an organic lipophilic solvent. Both monomers react at the droplet interface to form a polymeric membrane, the shell of the microcapsule. The core material can be hydrophobic or hydrophilic. Four main types of polymers are used for shell synthesis by this method: polyamides (reaction of diamines and bidentate chlorides), polyurethanes (reaction ofdiisocyanates and diols), polyureas (reaction of diamines and diisocyanates), and polyesters (reaction of bidentate chlorides and diols). The formation of polymer shell architecture at the interface involves complex unexplored mechanisms.

The main methods of interfacial polymerisation involve working with the following systems: liquid-solid, liquid-liquid, or liquid-in-liquid (emulsions), monomers can contain either one or both liquid phases. The technology has an important disadvantage of non-equilibrium process, which complicates the control and affects the course of the reaction.

Chemical methods also include the synthesis of polyelectrolyte nanocapsules: the "Layer-by-layer" method - electrostatic self-assembly, which was proposed by scientists of the Max Planck Institute in 1998 [56]. The "Layer-by-layer" (LbL) method was first used to form monolayer ultrathin polymer films on a macroscopic substrate. In 1966, the authors of [57] proposed to use alternating adsorption for film assembly. In 1991, Decher et al. reviewed a method for the preparation of polyelectrolyte films, which consists of one-by-one adsorption of polycations and polyanions on a substrate [58].

In 1998, the technology was successfully transferred to surface nanoengineering of micron and submicron-sized nuclear particles by alternating polyelectrolytes having opposite charges [59]. Although electrostatic interactions are the main driving force of LbL deposition, polyelectrolyte capsules are also synthesised based on covalent [60-61], dispersion [62], and guest-host interactions [63], which exhibit unique properties.

Polyelectrolyte shells can be formed on both colloidal particles and templates. These can be inorganic or organic particles ranging in size from 20 nm to tens of microns, micro- and nanocrystals of drugs or dyes, compacted DNA, protein aggregates and biological cells [64]. Calcium [65] and manganese carbonates [65], calcium phosphate [66], silicon oxide particles [67], polymer microparticles, for example, based on melamine formaldehyde resin [68] are used as templates.

A capsule shell of different architecture is formed on templates. It is determined by the properties of oppositely charged polyelectrolytes, formation conditions, and the number of layers. It is also possible to synthesise shells with different thickness and morphology by varying the conditions of shell synthesis: pH, ionic strength and nature of polyelectrolytes, their concentration, charge density of the polymer chain and molecular weight. Natural (polysaccharides, chitosan, gum) and synthetic polyelectrolytes (polydimethyldialylammonium chloride, polyacrylic acid, polystyrene sulfonate, polyallylamine, etc.) are used in encapsulation processes. Their choice depends primarily on the purpose of the encapsulated substance. Natural biocompatible polymers are used for medical purposes [69], and synthetic polymers for technical purposes [70].

Once the shell is fully formed, the templates are most often removed by dissolution with substances which properties are determined by the chemical properties of the particle. If degradable templates are used, the nuclei can be removed not only by dissolution, but also by decomposition with the use of proper physicochemical methods, pH change, heat treatment. Weakly cross-linked melamine formaldehyde (MF) particles were initially used most intensively as matrices for the fabrication of hollow polyelectrolyte capsules. However, they dissolve at low pH or in water-miscible organic solvents such as dimethyl sulfoxide. Incomplete removal by MF of oligomers formed during dissolution and their biohazard have severely limited the applicability of these nuclei [71]. Biodegradable polymeric microparticles, such as polylactic acid copolymers, easily overcome the disadvantages of MF but pose other limitations such as polydispersity and tendency to aggregate [72]. Conventional inorganic cores such as silicon oxides can be completely dissolved, but with the dangerous hydrofluoric acid only [73].

Nevertheless, even biological cells, such as red blood cells as matrices for LbL assembly, are not veryappropriate because the removal of the nucleus occurs using strong oxidising agents [74]. Each of these considered templates has its own application limitations and is not well suited for LbL technology.

Calcium carbonate is the most environmentally friendly and is considered a safe material to introduce into a biological system. It has been proven to be completely removed without any residual elements in polyelectrolyte capsules [73].

When $CaCO_3$ particles are used as degradable templates for the assembly of multilayer polyelectrolytes using the LbL method, the subsequent removal of the template is performed by acetic acid or a chelating agent, resulting in the complete removal of calcium carbonate from the core [75].

Ethylenediaminetetetraacetic acid (EDTA) is more commonly used to dissolve calcium carbonate [76]. The released volume is filled with a functional substance. This encapsulation technique is suitable for water-soluble functional substances such as proteins, biologically active compounds, and pharmaceuticals [77].

The process of capsule formation on colloidal particles uses aromatic essential oils [78], fat-soluble vitamins [79], medicinal substances [80], and repellents [81] as functional substances.

The authors of [82] provide the following classification of microcapsule applications for functionalisation of textile materials and products (Fig. 2). The application possibilities of microencapsulated textile auxiliaries are quite wide: from materials characterised by one simple function to imparting polyfunctional properties to fabrics.

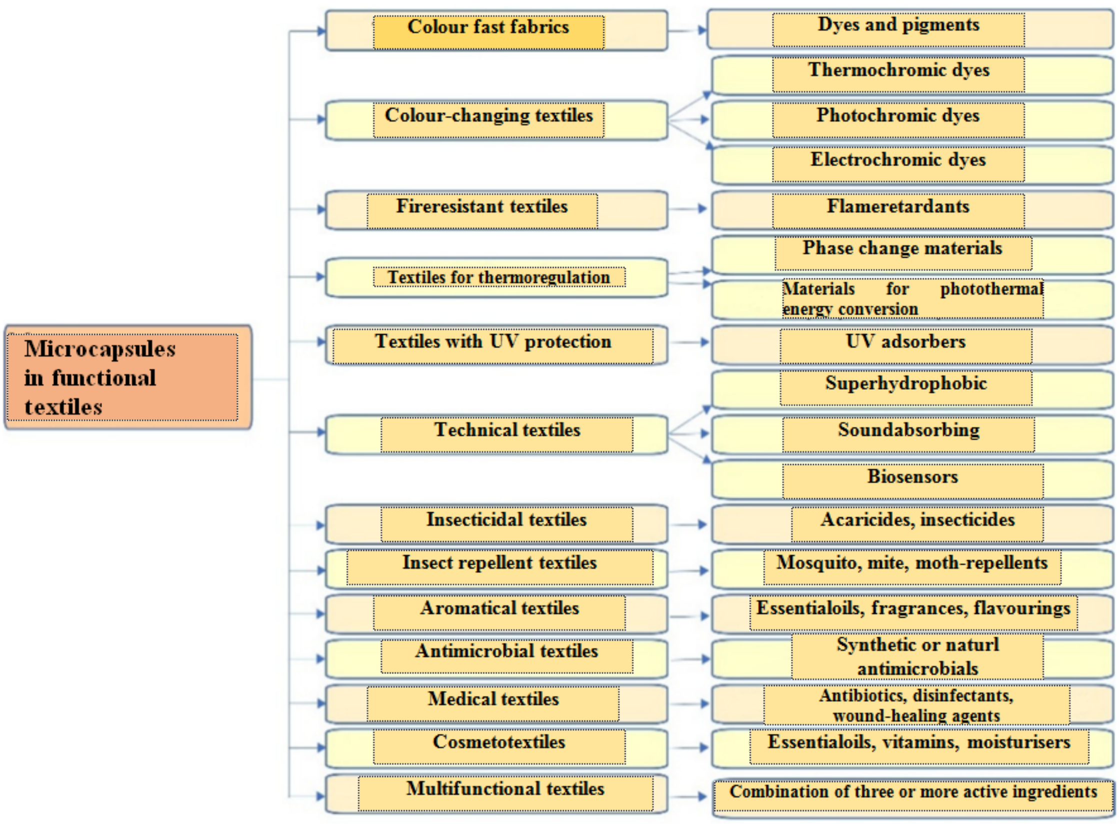

Fig. 1. Using microcapsules for textile functionalization [82]

Natural focal infections transmitted by ixodal ticks (tick-borne transmissible infections - BTIs) are a serious public health problem. The reasons are: their widespread expansion on the territory of Russia and in the world, the mass incidence of diseases, etiological diversity, high frequency of mixtforms, severity of course and outcomes, increasing number of anthropurgic foci in suburbs and on the territory of cities. Population or individual defence methods protecting humans from contact with vectors, and therefore from any vector-borne infection, are not widely used for a number of reasons [83-85].

The dangerous disease can be prevented by several preventive measures: anti-tick treatment of dangerous areas in endemic territories, vaccination of the population against tick-borne viral encephalitis (TSE), emergency immunoprophylaxis for persons affected by tick bites, and the use of protective equipment against ticks [86].

We have developed an encapsulation technology for acaricide-repellent preparations. The capsule core includes alphacypermethrin dissolved in non-toxic oil solvent. The capsule shell architecture is made of polydimethyldialylammonium chloride and acrylic copolymer of acrylic, methacrylic, maleic acids, and their salts. We obtained a stable dispersion of microencapsulated acaricide-repellent preparation with particle size from 30–300 nm depending on the number of layers in the shell composition [87]. Suits treated with such a preparation ("Barrier-Insecto U") have been tested in the conditions of the natural focus of viral tick-borne encephalitis in natural biotopes of the Irkutsk region and Khanty-Mansiysk Autonomous Okrug. They provide high protection against tick vectors (protective action coefficient (PAC) against ticks = 98.2%) [88, 89].

Microcapsules with essential oils or flavouring compounds of other nature included in the core are used for flavouring of textile materials. Such microencapsulated preparations are able to gradually release active ingredients through permeable shells [90]. If the shell is formed by interfacial polymerisation, the release of the fragrant substance occurs as a result of mechanical action, for example, by friction during wear of the product. The method of immobilisation of filled capsules on textile material plays an important role for creating permanent fragrant finishes [91-93].

Most aromatic substances have several beneficial properties including therapeutic, pharmaceutical and antimicrobial ones [94]. Citrus, lavender, rose or vanilla fragrances encapsulated in fabric lead to well-being and have important psychological and emotional significance for humans, while peppermint and lemon oil have a high sedative effect, lavender and rosemary contribute to a person's recovery from influenza. The use of natural encapsulated oils immobilised on textile material is beneficial to human health, and is characterised by minimal negative effects [95]. Aromatherapy type of finishing is proposed for bed linen, sportswear, socks, women's textile accessories. Aromatherapy is the most well-known and widely used method among alternative approaches to the treatment of diseases [96].

Textile materials containing substances with a variable phase state capable of exhibiting thermoregulating properties in a certain temperature range are of particular interest. One of the most actual ways to impart thethermoregulating properties to textiles is the application of special coatings on the surface of fibres or products, which is based on the use of a composition containing microcapsules containing substances that have the ability to phase transition [97].

These include paraffin waxes and linear chain hydrocarbons, which have the ability to store heat and are capable of phase transition at temperatures close to human body temperature. Furthermore, some naturally occurring organic oils as well as fatty acids are not only substances capable of storing and releasing heat under certain conditions, but also have many other useful properties. These advantages make them effective for the production of microcapsules based on them and applications for imparting thermoregulatory properties to textile materials [98, 99].

For instance, for encapsulation it was proposed to use coconut oil, which has the ability to phase transition. This substance is available and inexpensive, safe for the skin, harmless to the environment, etc. It makes it favourable for introduction into the core of the microcapsule [100, 101].

For obtaining textile materials with a long-term thermo-regulating effect resistant to washing and other chemical-physical effects, the appropriate design of shell-forming components is an important aspect of the microencapsulation process. It is necessary to select substances which ensure a high shell strength to prevent premature release of the active ingredient from the capsule on the textile material, which could reduce the finishing technical results. The use of urea and low-formaldehyde precondensates of thermosetting resins (Otexid D-2, Otexid NF), which also act as microcapsule stabilisers, has been proposed. We have selected acidic catalysts providing a more complete course of the shell formation reaction (acetic acid, magnesium chloride 6-aqueous). We tested auxiliary stabilisers for the obtained microcapsules: PVA and salicylic acid, which are the most widely known in the production of medical preparations. We used Tween 80 for emulsification of coconut oil, which is used in biomedical development and cosmetology [102, 103].

There are several methods of applying microcapsules to textile materials. One of the most common methods is the incorporation of the capsules into the fibre during the electrospinning process. Two methods of immobilising microcapsules onto textile material are more accessible in the finishing industry: by impregnation in the resulting dispersions or by printing. The impregnation and printing compositions with inclusion of microcapsules based on acrylic thickener and Ruzin-14I binder are effective [104, 105]. These application methods are easy to implement and are cost-effective.

Conclusion

Nanotechnology and nanoparticles as well as nano- and microcapsules are widely used in the global practice of fibres, textiles, and special purpose products. The nanotechnology principles make it possible to produce textiles and textile products with special properties for different areas of use: home textiles, clothing, army uniforms, medical textiles, sports textiles, technical textiles, etc.

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