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BEND SENSORS BASED ON NANOCELLULOSE AND POLYVINYL ALCOHOL BIONANOCOMPOSITES FOR WEARABLE ELECTRONICS

Introduction. *Currently, artificial polymers that pollute the environment are used in bend sensors. Nanocellulose (NC) is a biodegradable and flexible material, but it has a low elongation ability, which limits its use for human motion detection. Creating NC-based composites is a way to solve this problem.*

Problem Statement. *Synthesizing bend sensors based on biodegradable material (bionanocomposite of nanocellulose (NC) and polyvinyl alcohol (PVA))) to be used in sensors for analyzing human muscle activity is an urgent problem.*

Purpose. *To determine the effect of the sensor substrate material on the operating parameters of bend sensors.*

Materials and Methods. *The synthesis methods have been as follows: acid hydrolysis of organosolvent cellulose to obtain NC, vacuum casting to obtain NC-PVC nanocomposite films, and high-frequency magnetron sputtering to produce strain-sensitive films. The following research methods have been employed: optical spectrometry, mechanical elongation and tensile testing, souil burial degradation test, and strain measurement.*

Results. *NC-PVC composites have been synthesized and bend sensors have been created on their basis. The main electrical parameters of the obtained bend sensors are as follows: the strain sensitivity coefficient is 7.52, the reversibility ranges within 9—23%, the time drift varies within 0.17–0.5%/min. The biodegradability of the composite is 21—70% mass loss in 4.5 months. The effect of the sensor substrate material on the functional properties of these sensors has been investigated. It has been found that the addition of PVA to NC improves the optical and mechanical properties of the composites.*

Conclusions. *The optimal composition of the composite can be considered a mix of NC-PVC in a ratio of 1 : 1. The developed bend sensors can be used to monitor human muscle activity for medicine, sports, and rehabilitation.*

Keywords: bend sensor, nanocellulose, PVA, bionanocomposite, biodegradability, and wearable sensor.

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Wearable sensors are one of the most promising areas in modern electronics. Today, many different types of wearable sensors have been developed, including humidity sensors [1], temperature sensors [2], glucose sensors [3], bend sensors [4], light sensors [5], sensors for environment monitoring [6] and so on. Strain sensors have become one of the most important elements used for evaluating the human body muscular activity. These sensors respond to pressure, stretching, bending, twisting, and other influences. In particular, bend sensors are used for monitoring human motion (walking and running) when they are attached to joints, as well as for monitoring human pulse and blood pressure when attached to the skin near blood vessels [7]. Additionally, they are used for monitoring swallowing and speech when attached to the larynx [8], and for monitoring facial expressions when attached to the face [9]. The areas of application for these devices include medical devices, robotics, and household electronics.

Currently, artificial polymers such as polyacrylic acid (PAA) [10], polyacrylamide (PAM) [11], polyethyleneimine [12], polyamine [13], polyamide [14], and polyoxyethylene [15] are used in bend sensors. However, these materials have their drawbacks, including environmental pollution, the need for disposal, and low biocompatibility. The use of biopolymers is quite promising, as they are made from natural and environmentally friendly materials such as cellulose, sodium alginate, starch, chitosan, proteins, and their derivatives.

Nanocellulose (NC) is a type of cellulose with one-dimensional crystal or fibers sized less than 100 nm. Such material is characterized by high aspect ratio, large specific surface area, high mechanical strength, and hydrophilicity. The ability of NC to decompose in the environment makes it ideal for use in disposable devices, i.e. devices that do not require disposal process. Additionally, NC is a biomaterial obtained from plant sources (cotton [16], flax [17], hemp [18], bamboo [19], reed [20], miscanthus [21]), making its production process environmentally friendly. The modifiability of nanocellulose is another unique property that is explained by its three-dimensional network structure, where the structural units are connected by physical entanglement, physical stitching (hydrogen bonding, electrostatic interaction, Van der Waals force), or chemical stitching (covalent bond, ion binding, polymerization) of monomers. Such a structure enables to hold a large amount of water or enhance interaction with small molecules, polymers, and nanoparticles, which facilitates the fabrication of functional composites based on it. One of the most significant drawbacks of nanocellulose is its low elongation that limits its use for evaluating high-amplitude movements. Additionally, nanocellulose has high hydrophilicity. This fact can lead to a deterioration in its mechanical properties under high humidity or contact with water. One way to address these issues is to create composites based on nanocellulose with other materials that complement its properties. For example, adding polymers with a high elastic modulus can increase the composite's ability to stretch, while adding hydrophobic coatings can reduce its hydrophilicity.

Nanocellulose and its composites are used in bend sensors in various morphological forms, such as films [22] and gel-like materials (hydro- and aerogels) [23]. Nanocellulose films are thin layers of nanocellulose with a thickness of 20—100 μm, which can be used as separate substrates onto which a strain-sensitive thin film is deposited. This form of nanocellulose has the advantage of a planar device construction and flexibility, but its disadvantages include low stretchability and sensitivity, which are determined by the material of sensitive layer, typically of metallic nature. Hydrogels are two-phase polymer materials consisting of a porous solid part and a significant amount of water, forming a dense gel-like mass [24]. The advantages of this form of NC composites in bend sensors are their high stretchability and high sensitivity [25] due to the fact that the polymer with ionic conductivity is the sensitive layer and high-elastic substrate simultaneously. However, this form of the sensitive layer cannot be used in an integrated sensor, as it is characterized by bulky, an uneven and non-planar surface.

This study involves synthesizing a bio-nanocomposite in the form of a planar film based on pure nanocellulose and polyvinyl alcohol (PVA) that provides the high elasticity of the composite. Existing developments of similar materials today mainly concern the study of physical properties of the composite itself and to a lesser extent are dedicated to their device application, including strain sensors [26]. The issue of developing of an optimal strain-sensitive element on the surface of such composites in order to create highly effective bend sensors remains unresolved. The aim of this study is to determine the influence of substrate material (pure NC, pure PVA, and a composite based on them) on the parameters of bend sensors.

Synthesis of NC-PVA composites. Nanofibrillated cellulose is extracted by hydrolysis of non-dried organosolv cellulose from miscanthus (*Miscanthus giganteus*) stems with a sulfuric acid solution with a concentration of 43% at a temperature of 60 °C for 60 min and ultrasonic treatment to obtain a transparent, stable nanocellulose suspension over time.

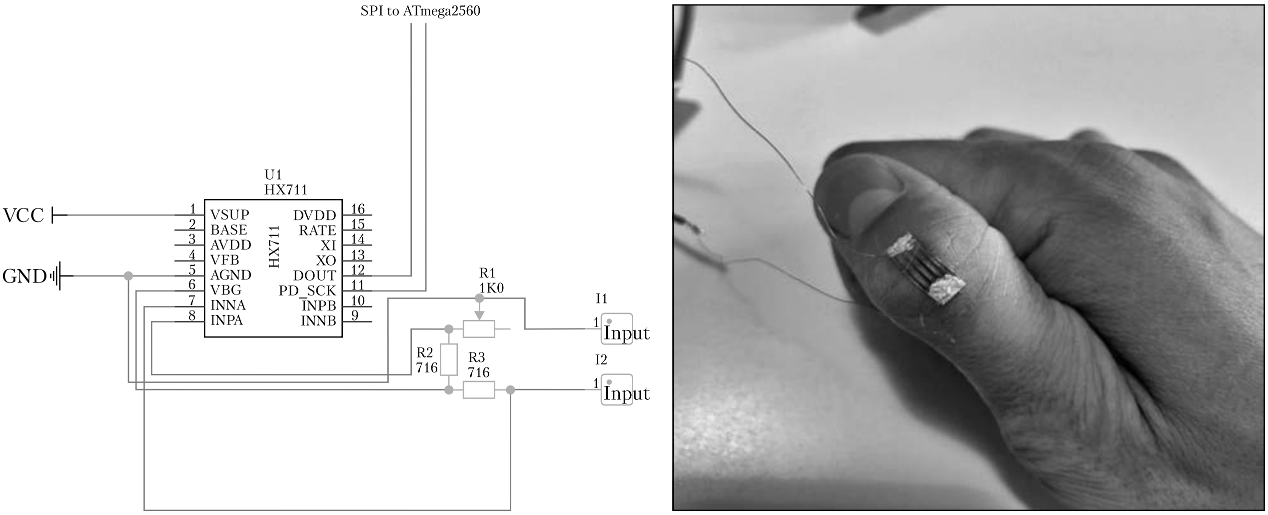
The manufacturing process of a solution of polyvinyl alcohol (PVA) involves dissolving of 1 g PVA granules in 100 mL of deionized water at a temperature of 90 °C while stirring constantly for 30 min. Liquid composites are made from solutions of nanocellulose and polyvinyl alcohol, mixed in different concentrations that are expressed in mass percent (% (w/w)). The following composites have been obtained: 0% (w/w) PVA (pure NC), 50% (w/w) PVA, 75% (w/w) PVA, and 100% (w/w) PVA. Then the obtained solutions are subjected to air removing by vacuum, after that each suspension is poured into Petri dish and dried in a thermal chamber at a temperature of 60 °C until a continuous thin film is formed (*vacuum-assisted casting*). The thickness of the composite film varies as the mass of the suspension in the Petri dish chnages. As a result, the thickness of obtained films ranges from 35 to 450 μm.

Fabrication of strain sensors based on NC-PVA composites. Obtained bend sensors consist of a two-layered Ti-Ni film, deposited onto the surface of NC-PVA nanocomposite film, and attached leads. The metal piezoresistive film is deposited by reactive RF magnetron sputtering in an argon environment. The voltage during the sputtering process is 600 V, the current is 1 A, the pressure in vacuum chamber is $5 \cdot 10^{-3}$ mmHg, and the deposition temperature is 50 °C. Thin Ti layer of 30 nm thickness is used to improve the adhesion between inorganic (Ni layer) and organic (NC-PVA composite) materials. The thicknes of Ni layer ranges within 200—250 nm. The U-shape of piezoresistive film is defined by the corresponding pattern into the magnetic technological mask. Next, the nanocomposite film with deposited strain elements is cut on separate samples. Sensor leads are attached by conductive paste with silver particles.

Characterization Techniques. Optical transmittance spectra of NC-PVA composite films are measured by 4802 UV/VIS Double Beam Spectrophotometer in the range from 190 to 1100 nm. Mechanical strength tests of obtained sensors have been performed by means of vertical breaking machine RMB –30—2 M.

To evaluate the biodegradability of bend sensors made from NC-PVA composites, a soil burial degradation test has been conducted. Sod-podzolic soil from the flower bed at NTUU "Igor Sikorsky Kyiv Polytechnic Institute" has been used. The samples are buried at a depth of 20—30 mm. Mass measurements are carried out on a half-month basis, while the temperature remained at 19—21 °C. To maintain humidity, the soil with samples is sprayed with water every 3—4 days. The mass of the samples is measured by high-precision digital scales with a built-in level EDIS 50 (50/0.001 g). Samples are cleaned from soil residues by a dry brush made of squirrel hair.

The electrical properties of bend sensors have been studied by the Power Supply HM8143 and the MS8040 Multimeter. Also, hand-made measuring equipment has been developed to inves-



a *b*

Fig. 1. Electrical schematic diagram of connection the bridge to the ADC (*a*) [4] and image of attached strain-sensitive element to the human skin (*b*)

tigate the piezoresistive characteristics of bend sensors [4]. This measuring equipment consists of a mechanical and an electrical part. The mechanical part is a frame with a micrometer screw, on the top of which a plate of high-alloy steel is fixed. The micrometer screw is used to bend the sample. The bending magnitude is controlled by a micrometer indicator head.

The electrical part of the measuring equipment has the following components: Wheatstone bridge, analog-to-digital converter (ADC) HX711, microcontroller ATmega2560, UART-TTL converter CH340G, and Apple MacBook Pro Retina 15 laptop. In this study, the indirect measurement method by a Wheatstone bridge has been employed to measure small changes in the sensor resistance during bending. The Wheatstone bridge is an electrical circuit consisting of four connected resistors. One of them is strain gauge based on NC-PVA composite, which changes its initial value during bending, while the other resistors remain unchanged. Change in the resistance results in a change in the bridge output voltage that is measured by the ADC. To measure the bridge voltage shift, ADC HX711 from AVIA Semiconductor is chosen. It is a 24-bit analog-to-digital converter with high accuracy, used to measure small voltage shifts. This chip contains two internal programmable amplifiers that allow determining the smallest voltage changes. Moreover, the HX711 has an SPI interface for data transmission to microcontrollers, which makes it easy to use. The ATmega2560 microcontroller is employed to interact with the ADC and perform calculations. By connecting the Wheatstone bridge to the HX711 (Fig. 1, *a*), it is possible to obtain very accurate measurements of the voltage shift on the bridge, followed by calculation of the resistance based on the bridge resistor parameters.

During testing of the bend sensors for evaluation of human muscle activity, the strain-sensing element is attached to the skin by medical adhesive BF-6 on the elbow or thumb (Fig. 1, *b*). A changeable pad and sticky medical tape are used to fix the sensor leads to the skin in order to reduce mechanical stress on the contacts.

To obtain more detailed information about the surface morphology of different substrates and their structural heterogeneity, we have employed the atomic force microscopy (AFM) method. These investigations have been conducted by means of NT-MDT Solver Nano microscope in semi-contact mode by a Cont-AC microcantilever with a radius of curvature less than 10 nm. The AFM images have been analyzed by the NT-MDT Nova software that provides advanced image processing for extracting quantitative data from the AFM images (surface roughness and feature size). Root mean square (r.m.s.) of surface roughness is determined for a scanning area of 60 × 60 μm. Also the surface morphology of Ni thin film has been investigated by scanning electron microscopy (SEM) using a REM-106U microscope in secondary electron mode and high vacuum.

Optical, mechanical and biodegradable properties of NC-PVA composites. The appearance of obtained NC-PVA composites is shown in Fig. 2. As can be seen from the above photos, bend sensors based on NC-PVA composites have a flat shape and are quite transparent. At the same time, the transparency (T) of the composites increases in the direction from pure NC to pure PVA. To quantify the transparency of the composites, their optical transmission spectra have been measured (Fig. 3). It has been found that the least transparent sample is a pure NC, and the most transparent is a pure PVA: the transparency coefficient at a wavelength of 600 nm is 22 and 92%, respectively. An increase in the amount of nanocellulose in NC-PVA composite reduces the transparency of the material. In particular, by adding 25% (w/w) (or 50% (w/w)) of NC, the transparency of PVA decreased by 18% (or 34%). This is probably due to the increased light scattering caused by the presence of nanofibrils in pure nanocellulose. So, the addition of PVA to NC significantly increases its optical transparency. So, it can be used for transparent strain sensors.

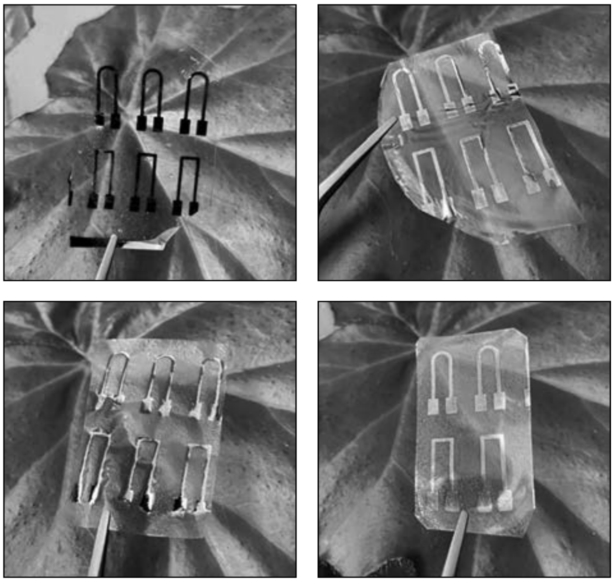


Fig. 2. Images of bend sensors based on NC-PVA composites (PVA content of 100% (w/w), 75% (w/w), 50% (w/w), 0% from left to right)

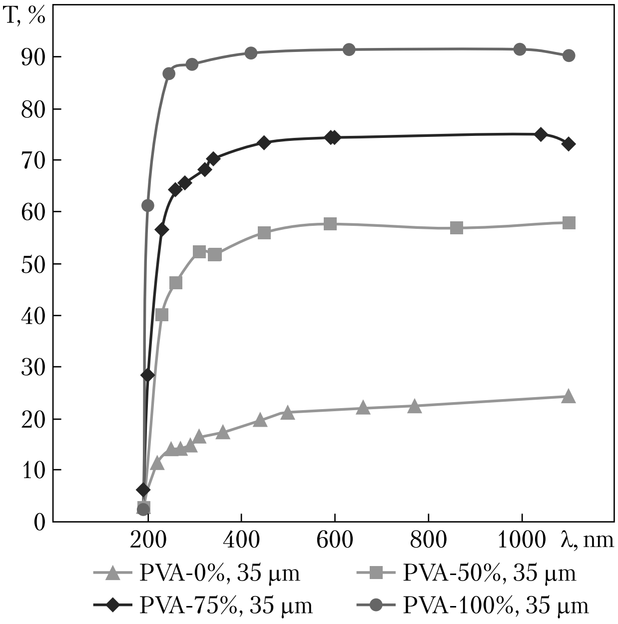
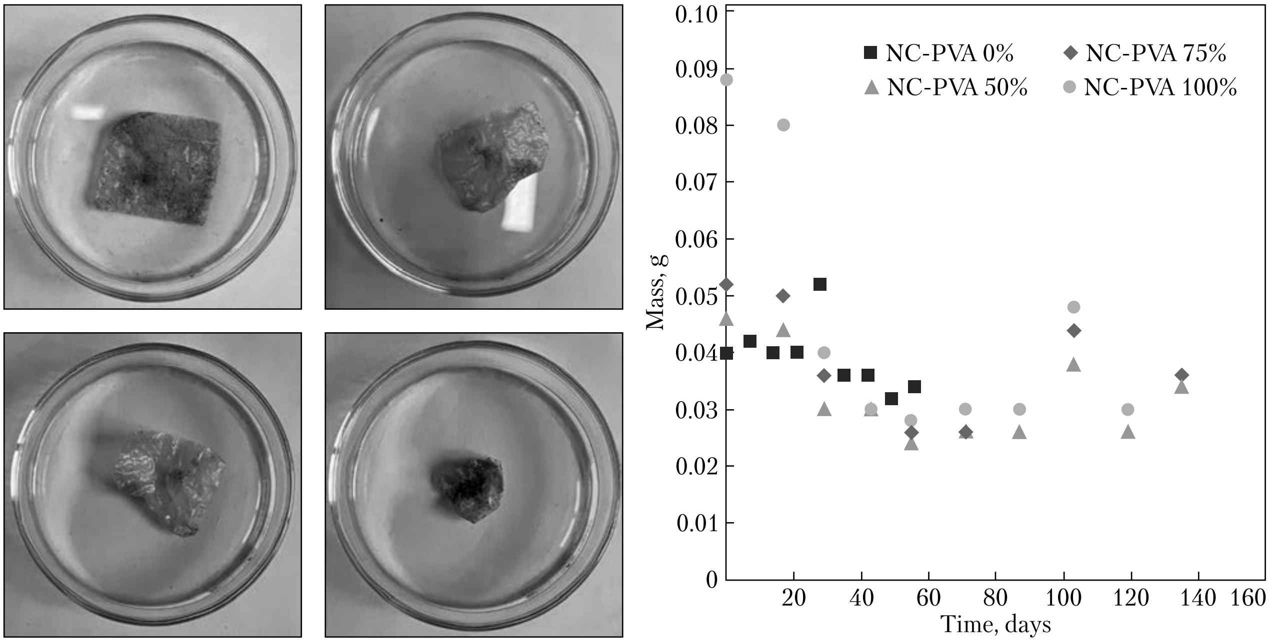


Fig. 3. Optical transmission spectra of NC-PVA composites

Also mechanical properties of NC-PVA composites have been investigated. It has been shown that thickness of the composites has a significant effect on their strength and elasticity. When the thickness decreases more than 12 times, the elongation decreases 4 times, and the breaking force decreases 2 times. Also, it has been determined that the adding of 50% (w/w)NC to the composite resulted in an increase in material strength by more than 10% at the same thickness.



a *b*

Fig. 4. Evaluation of biodegradability of NC-PVA composites based on images (*a*), the graph of the dependence of the sensors mass on the time spent in the soil (*b*)

This can be explained by the fact that PVA is reinforced with NC nanofibers. The maximum breaking force (66 MPa) and elongation (22.5 mm) have been reported for the composite of NC-PVA 50% (w/w) at a thickness of 330 μm. Thus, the application of NC-PVA composites makes it possible to achieve increased strength and elasticity of strain sensors.

To evaluate the biodegradability of obtained nanocomposites, soil burial degradation test has been performed during 4.5 months. For this purpose, samples of equal square shape (30 × 30 mm) are cut out. Visual observation of the samples shows that they decompose in the soil with changing of their shape and size (Fig. 4, *a*). The calculations of the mass loss in 4.5 months of soil burial degradation test have shown that PVA material lost 70% of its mass, while composites NC-PVA 75% (w/w) and NC-PVA 50% (w/w) lost 63% and 30% of their mass, respectively (Fig. 4, *b*). According to previous studies, a pure NC lost 21% of its mass in a similar period of time. PVA material decomposes more quickly in soil than nanocellulose. When water and microbes are introduced to PVA in soil, it undergoes a transition from a polymer to individual monomer units that subsequently undergo biodegradation. Thus, the addition of PVA to nanocellulose improves its biodegradability.

The surface morphology of used substrates can be seen from the AFM images in Fig. 5. PVA exhibits a relatively smooth surface, but it contains cavitation air voids (dark spots in the image). The maximum height difference is 65 nm and r.m.s. roughness is 27 nm. The surface of NC contains nanoparticles and their clusters (bright spots), which increase its roughness up to 98 nm, with a height difference of 600 nm. The composite material containing 75% (w/w) PVA shows the presence of both types of structural irregularities: cavitation air voids from PVA and NC nanoparticles. The r.m.s. roughness is 102 nm, and the height difference is 135 nm. The composite material with 50% (w/w) PVA exhibits strong structural inho-

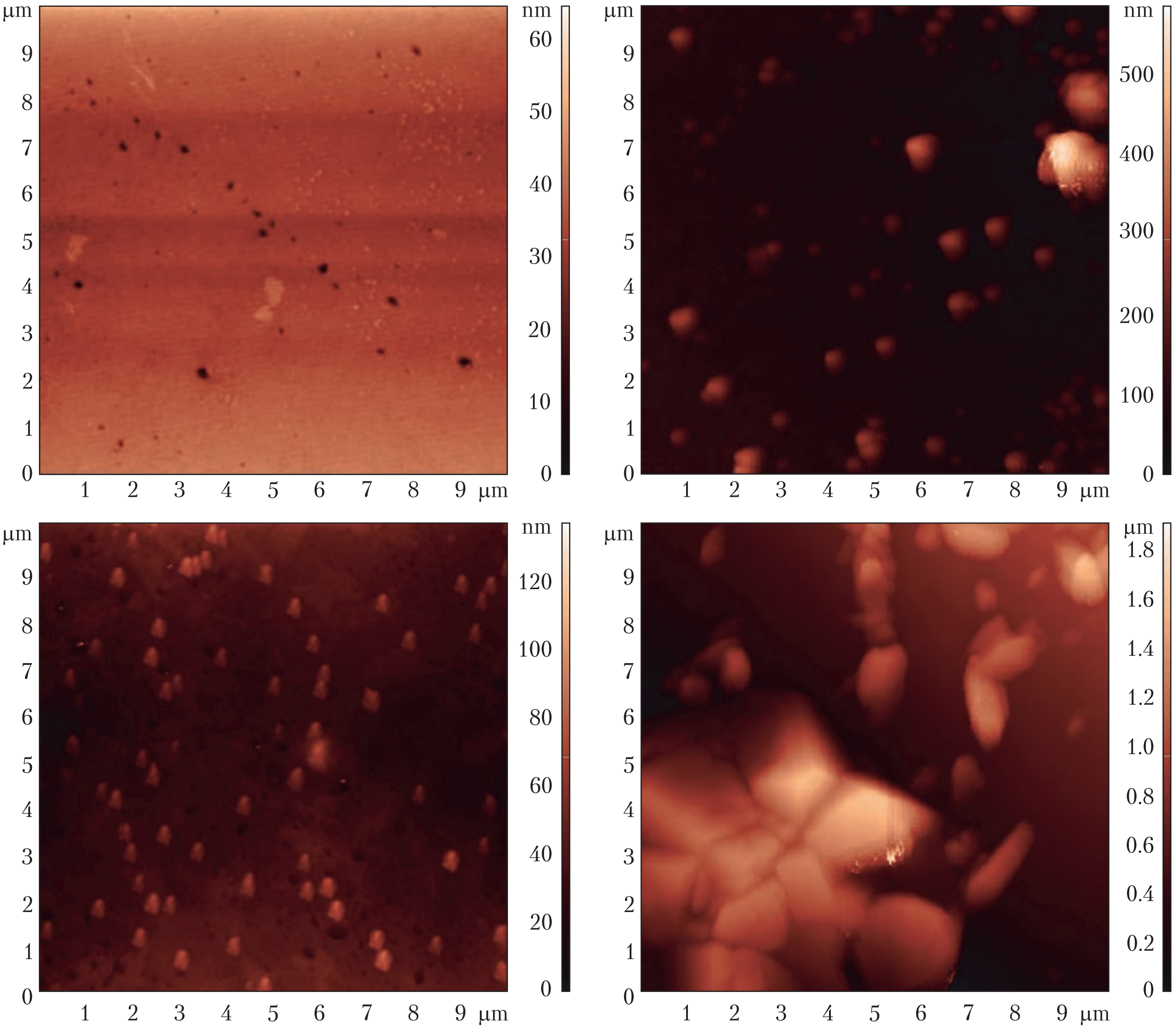


Fig. 5. AFM images of substrates morphologies (from the left to the right: NC-PVA 100% (w/w), NC-PVA 0% (w/w), NC-PVA 75% (w/w), NC-PVA 50% (w/w))

mogeneity due to the clustering of NC nanoparticles in separate flakes. The r.m.s. roughness is 346 nm and the height difference is 1850 nm. It is known that the structural inhomogeneity of material mixtures is always greater than that of pure components, reaching its maximum at approximately a 50 : 50% (w/w) composition, as observed in this study. Obviously, such a rough surface affects the structure of the metal film on its surface. Figure 6 shows microcracks in the structure of the thin metal film as seen in the SEM image.

Electrical and piezoresistive characteristics of strain sensors based on NC-PVA composites. A piezoresistive effect has been observed for all obtained sensors: with an increase in linear elongation, the resistance of sensors increases (Fig. 7). As can be seen from Fig. 7, the dependences of sensor resistance on linear extension

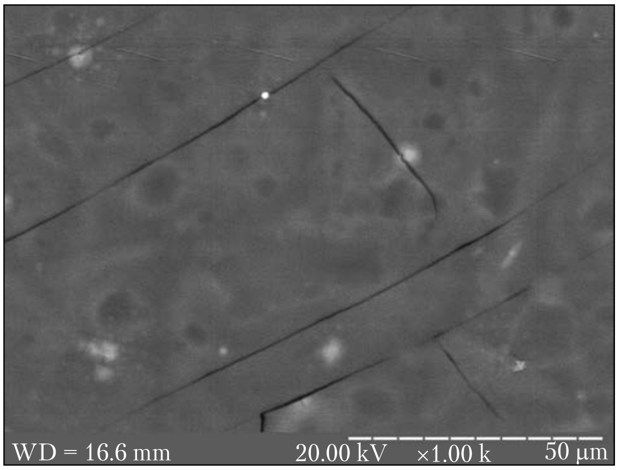


Fig. 6. SEM image of Ni thin film on NC-PVA 50% (w/w) substrate

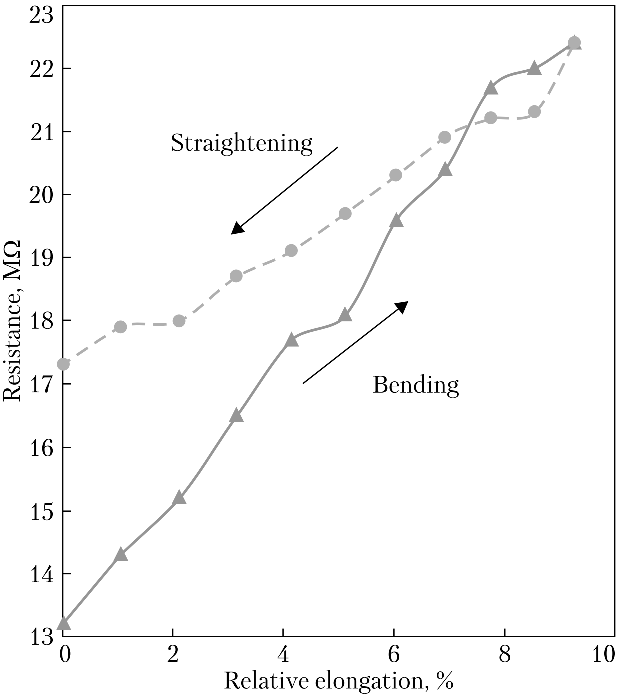


Fig. 7. Dependence of electrical resistance on the relative elongation based for bend sensors based on NC-PVA 50% (w/w) composite

under bending and straightening are approximated by a linear law. Some deviation from linearity is apparently due to the structural heterogeneity of Ni thin film on the surface of biopolymer substrate. The effect of NC-PVA composition on the initial resistance of the metal film has been studied. On the surface of pure NC, the strain gauge resistance is 2—3 kΩ, while on the PVA surface, it is ~0.4 kΩ. This is due to the fact that on smooth surface of PVA, the nickel film is deposited more homogeneously in structure. The strain gauges on the surface of the composites, on the other hand, have a much higher resistance (~37 kΩ for NC-PVA 75% (w/w) and ~13000 kΩ for NC-PVA 50% (w/w)).

The resistance of metal films depends on the roughness of the substrate that is commensurate or greater than thickness of the sensitive film [27—29]. Surface roughness can influence on both the increase in resistor length due to an elongation of the conductive pathway as has been studied by Siegel et al. [27, 28] for rough paper surfaces, and on the reduction in cross-sectional area of the film on the side surfaces of the irregularities as has been shown in [28]. The presence of structural inhomogeneities also affects the resistivity of the thin film [28]. In particular, structural inhomogeneities can be microcracks that significantly increase the resistance of the film, as shown in [29] and can be seen from SEM images (Fig. 6).

The sensitivity of the devices is also affected by the composition of the substrate. Coefficient of strain sensitivity or gauge factor (GF) of obtained sensors is in the range from 3.02 to 7.52. GF is calculated as the ratio of the relative change of resistance to the relative elongation of piezoresistive film.The strain sensitivity for devices on the surface of pure NC is 3.02, and on the surface of pure PVA is 7.1. For composites, the GF value is improved concerning pure NC and pure PVA. In particular, for the NC-PVA 50% (w/w) composite, the GF value is 7.52. At the same time, a further increase in the concentration of PVA in the composite worsens the GF value (3.27). The sensitivity of the strain sensors is influenced by both the roughness of the substrate (NC) and the plasticity of the material (PVA). A higher roughness leads to increased sensitivity, as it results in greater

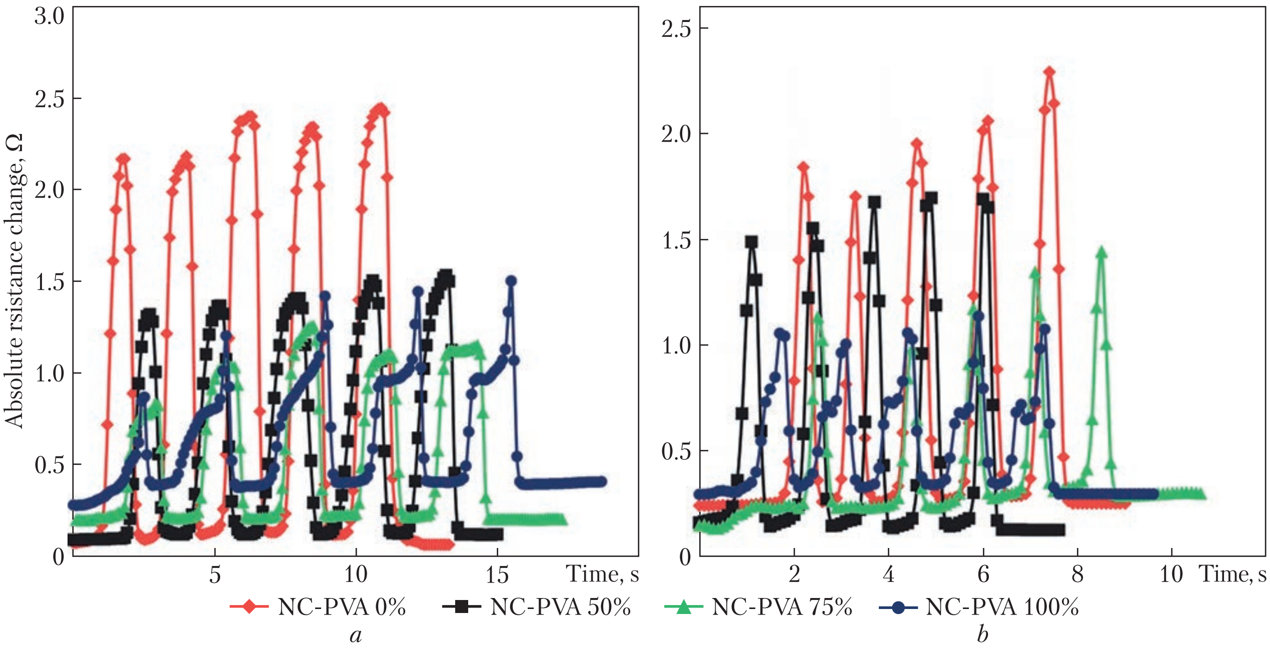


Fig. 8. Dependence of absolute resistance change on the time of bending-straightening cycles for elbow (*a*) and finger (*b*) with the use of obtained sensor based on NC-PVA composite

elongation of the strain resistor when samples are bent. On the other hand, PVA is a more elastic material than NC, so even on a perfectly smooth PVA, the GF is quite high. The maximum GF is achieved in the 50 : 50% (w/w) composites, which is attributed to the favorable influence of both the plasticity of PVA and the roughness of NC.

Reversibility of strain sensors is determined as the relative change of the resistance after one bending-straightening cycle. The reversibility of obtained sensors ranges from 9 to 23%. It has been found that for the sensor based on NC-PVA 50% (w/w) composite, there is a deterioration of recovery to the initial signal under straightening compared to the devices based on pure NC and pure PVA. Obviously, low reversibility is a result of large sensor response on this material.

The stability in time of obtained strain sensors is evaluated by means of drift coefficients. They are defined as the relative change in resistance during static bending of a given value per unit of time. In this study, the devices are subjected to the deformation with a relative elongation of 5% for 5 min. The device drift coefficient for pure PVA is 0.5%/min, while for pure NC it is only 0.17%/min. The NC-PVA 50% (w/w) composite has an average value of this coefficient between NC and PVA (0.38%). Deterioration of device stability in time can be explained by the greater elasticity of sensors based on NC-PVA composites comparing with pure NC.

Application of strain sensors based on NC-PVA composites for human muscular activity. Due to the significant variation in sensor recoverability, a short sensor configuration with better recoverability is used for measurements on the human body. The human muscular activity has been studied with the use of the obtained sensors placed on the skin near the place of maximum deformation of the limbs. In particular, finger and elbow bending and straightening have been measured. The sensors are fixed on the thumb joint and inside of the elbow joint. To evaluate their movement, the finger and the elbow are fully bent and stretched with the same frequency. Thus, the movement of the thumb or elbow joint can be converted into a cyclic change in sensor resistance in real time (Fig. 8). The sensors have shown sufficiently reproducible results at least after 5 cycles of bending-straightening. Slight deviations in the points of signal amplitude are caused by the fact that testing is carried out on a man. Therefore it is difficult to ensure identical movements, which leads to small fluctuations in the resistance. From Fig. 8 it can be concluded that the sensor on the pure PVA substrate has a significantly slower response than the sensor on the pure NC substrate (90 ms vs. 50 ms, respectively, in the finger test). With the addition of 50% NC to the PVA, the response time decreases and the signal magnitude increases.

Tensile test has been also performed for glued bend sensors on human skin under movement of finger and elbow before breaking. Bend sensor based on pure NC failed after 5—20 cycles of bending-straightening on the elbow and finger, respectively, while sensors based on PVA composites withstood more than 200 cycles.

In this study, NC-PVA bionanocomposites and bend sensors on their basis have been fabricated. The effect of the substrate material (NC-PVA 0% (w/w), NC-PVA 25% (w/w), NC-PVA 50% (w/w), NC-PVA 100% (w/w)) on the efficiency of these sensors has been investigated.

It has been found that the addition of PVA to NC improves the optical (transparency) and mechanical (elasticity) properties of the composites, as well as the strain sensitivity of the sensors based on them. However, the reversibility and stability over time of such sensors deteriorate. From this point of view, the mixture of NC and PVA in a ratio of 50:50% is the most preferable composition. The developed bend sensors can be used to monitor human muscular activity, since the addition of PVA significantly increases the possible number of bending-straightening cycles before breaking (10—40 times) and the device biodegradability.

The direction of further development is to study the effect of different configurations of strain-sensing element on the characteristics of NC-PVA bend sensors.

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ДАТЧИКИ ВИГИНУ НА ОСНОВІ БІОНАНОКОМПОЗИТІВ ІЗ НАНОЦЕЛЮЛОЗИ ТА ПОЛІВІНІЛОВОГО СПИРТУ ДЛЯ НОСИМОЇ ЕЛЕКТРОНІКИ

Вступ. На сьогодні в датчиках вигину використовують штучні полімери, які забруднюють навколишнє середовище. Наноцелюлоза (НЦ) є біорозкладним і гнучким матеріалом, однак має низьку здатність до видовження, що обмежує її застосування для оцінки рухів людини. Одним із способів вирішення цієї проблеми є створення композитів на основі НЦ.

Проблематика. Актуальним питанням є синтез сенсорів вигину на основі біорозкладного матеріалу (біонанокомпозиту із наноцелюлози та полівінілового спирту (ПВС)) для використання у сенсорах, що можуть бути застосовані для аналізу м'язової активності людини.

Мета. Визначення впливу матеріалу підкладки сенсора на робочі параметри сенсорів вигину.

Матеріали й методи. Застосовано методи синтезу: кислотний гідроліз органосольвентної целюлози для отримання НЦ, лиття з використанням вакууму для отримання нанокомпозитних плівок НЦ-ПВС і методу високочастотного магнетронного розпилення для виготовлення тензочутливих плівок. Методами дослідження були оптична спектрометрія, тестування на механічне видовження та розрив, тестування на біорозкладність у ґрунті, тензометрія.

Результати. Синтезовано композити НЦ-ПВС та на їхній основі створено датчики вигину. Основні електричні параметри одержаних сенсорів вигину: коефіцієнт тензочутливості досягав 7,52, реверсивність — 9—23 %, повзучість — 0,17—0,5%/хв. При цьому біорозкладність композиту становила 21—70% втрати маси за 4,5 місяців. Досліджено вплив матеріалу підкладки сенсорів на функціональні властивості цих датчиків. Встановлено, що додавання ПВС до НЦ покращило оптичні та механічні властивості композитів.

Висновки. Оптимальним складом композиту можна вважати суміш НЦ-ПВС у співвідношенні 1 : 1. Розроблені датчики вигину можуть бути використані для моніторингу активності м'язів людини, що є перспективним для застосування у медицині, спорті, реабілітації.

Ключові слова: сенсор вигину, наноцелюлоза, ПВС, біонанокомпозит, біорозкладність, носимий сенсор.