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EXPRESS METHOD FOR INVESTIGATING NATURAL WATER QUALITY USING A SENSOR DEVICE BASED ON SURFACE PLASMON RESONANCE AND A CONDUCTOMETER

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Background: One of the urgent contemporary issues is natural water pollution, which directly affects humanity's life support. This problem is associated with industrial and agricultural intensification and climate change. Water quality standards in Ukraine are defined by state standards, which regulate both organoleptic properties, such as turbidity and odor, and permissible concentrations of harmful substances.
Objective: The objective of this study was to develop a methodology for rapid natural water quality assessment using the SPR method and a conductometer and to simultaneously determine the durability of sensors with protective coatings.
Materials and methods: This study explores the feasibility of combining surface plasmon resonance (SPR) and conductometric methods to monitor the quality of natural water. The first stage involved modeling the concentration dependencies of SPR parameters and conductivity when adding controlled amounts of organic (sugar) and inorganic (table salt and soda) impurities to distilled water. Biological contamination was simulated using live yeast suspensions. Subsequently, samples of coastal water from the Dnipro River in Kyiv, the Stugna River near Vasylkiv, and a pond connected to the Stugna River near Borova village in Fastiv district were analyzed. All SPR studies were conducted using an improved sensor element with an additional protective zinc oxide layer, which reduced measurement errors typically associated with sensor replacement. To validate the reliability of the rapid assessment methods, water samples were additionally analyzed using standard laboratory methods at "Ukrkhimanaliz".
Results: The SPR results indicated that the Stugna River was the most polluted, followed by the pond, with the Dnipro River exhibiting the least pollution.
Conclusions: Summarizing the measurement results, it can be concluded that combining SPR and conductivity measurements enables rapid and objective assessment of natural water pollution levels. This corresponds to the total harmful impurities. Given the small dimensions and autonomy of the devices used in the developed methodology, river water monitoring can be carried out in field conditions by one person.
KEY WORDS: surface plasmon resonance; electrical conductivity; natural water control; natural water pollution; chemical analysis; optical properties.

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One of the urgent contemporary issues is natural water pollution, which directly affects humanity's life support. This problem is associated with industrial and agricultural intensification and climate change.

Water quality standards in Ukraine are defined by state standards [1–3], which regulate both organoleptic properties, such as turbidity and odor, and permissible concentrations of harmful substances.

Currently, many methods have been developed for monitoring, identifying impurities, and disinfecting water. Natural water contains a large number of insoluble substances, including sand particles, clay, silt, phytoplankton, and oxides of aluminum, iron, and manganese [4]. These particles affect water transparency. The first method for monitoring water turbidity was Jackson's candle turbidimeter [4], consisting of a candle and a cylinder. To measure turbidity, water was poured into the cylinder until the candlelight transformed into a shapeless spot. Turbidity in Jackson Turbidity Units was determined by the height of the water column.

Today, gravimetric, visual, and optical methods are used to study turbidity. The gravimetric method [5] is the most accurate but also time-consuming. It involves filtering a shaken water sample through a paper or membrane filter. The collected particles are washed with cold distilled water, dried, and the particle content is determined by the difference in the filter's mass before and after drying.

Optical methods for controlling the number of insoluble particles rely on light scattering on these particles. The nephelometric method [6] measures the intensity of light scattered by particles in a water sample, with particle concentration determined by comparison with a standard dispersive system. For rapid monitoring, a visual method [4] is used, where the color of water in a transparent cylinder is compared to a standardized sample.

The method of luminescence measurement can detect organic pollutants. When water is irradiated at specific wavelengths, peaks appear that correspond to various organic pollutants, such as chlorophyll [7]. Colorimetric sensors enable quick and selective pollutant detection through color changes resulting from chemical or quasi-chemical reactions with nanoparticles used as the sensor material [8, 9].

Chromatography allows for the quantitative detection of impurities, such as micro- and nanoplastics [10]. To identify pathogens, DNA-based biosensors, molecular biosensors, and fluorescent sensors are used [7]. Recently, machine learning and artificial intelligence technologies have been applied to analyze natural water quality [11, 12].

In recent years, special attention has been given to the implementation of innovative methods based on optical and chemical sensors, which provide rapid and accurate analysis [13, 14]. These approaches enable the detection of even microscopic pollutants, such as nanoplastics and traces of organic compounds resulting from anthropogenic activities [15].

Significant advances in machine learning and artificial intelligence technologies have made it possible to apply these technologies to water quality monitoring, significantly increasing the accuracy, objectivity, and productivity of monitoring [16–18]. The application of these technologies allows for the automation of impurity and contaminant detection processes, reducing analysis time and improving decision-making efficiency. For example, surface plasmon resonance-based methods are employed to assess water contamination levels [19]. Relevant studies have confirmed the effectiveness of these technologies in water quality control [20, 21], making them promising for implementation in water supply facilities.

MATERIALS AND METHODS

A new method for monitoring drinking water purification was developed and patented [22, 23], involving a surface plasmon resonance (SPR)-based device to assess the efficiency of water purification via freeze-thawing. These experiments were conducted on the “Plasmon-6” device

Fig. 1, developed by the V. E. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine. The main parameters of the device are presented in Table 1.

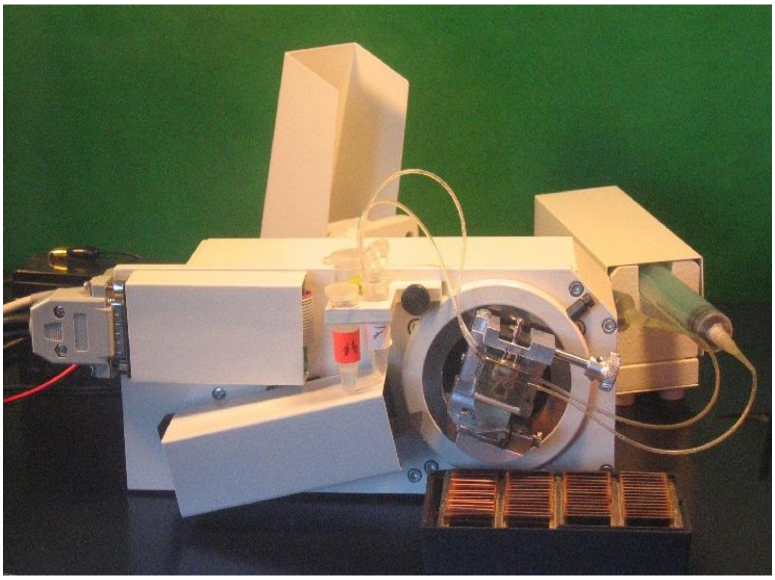

Fig. 1. Appearance of the "Plasmon-6" Device.

Table 1. Key technical specifications of the "Plasmon-6" Device

Parameter	Value
1	2
Baseline Noise (RIU) ang. sec	1.0–1.43
Absolute Measurement Error (RIU)	0.00005-0.0003
Surface Plasmon Resonance Curve Measurement Time	< 3 sec
Kinetics Measurement Speed	2 units/sec
GaAs Laser Wavelength	650 nm

Surface plasmon resonance is a phenomenon that occurs when free electrons in a thin metal layer are excited by incident light at a specific angle. In the case of gold, surface electrons collectively oscillate in response to light of a particular wavelength, producing peaks in the reflected light spectrum that do not appear in the incident light spectrum. This effect, occurring on the metal film surface, extends into the surrounding environment and decreases exponentially with distance. Molecular interactions on the surface influence the plasmon wave attenuation, resulting in changes in surface plasmon characteristics, such as shifts in resonance angle and alterations in the refractive index of the surface layer. This allows for real-time monitoring of chemical and biological reactions.

The device uses a prism-based method for plasmon excitation and operates in the Kretschmann optical configuration (Fig. 2) [24].

The "Plasmon-6" SPR device consists of a semiconductor laser, a glass prism, and a silicon photodiode. The sensor element, where plasmon excitation occurs, is positioned on the prism. Optical contact is maintained by immersion liquid. To obtain angular responses, the SPR sensor performs mechanical scanning by rotating the prism. The device operates as follows: the prism changes its position incrementally (driven by a stepper motor) within the range of total internal reflection angles at the prism-metal interface relative to the laser beam direction. During each scan, the reflection characteristics are measured, and the resonance angle is determined. The sensor response is displayed as an angular shift in reflection characteristics and a corresponding change in resonance angle due to variations in the refractive index of the medium in contact with the gold film. The measured resonance angle range spans from 38° to 69°, corresponding to refractive index changes from 1.00 to 1.5.

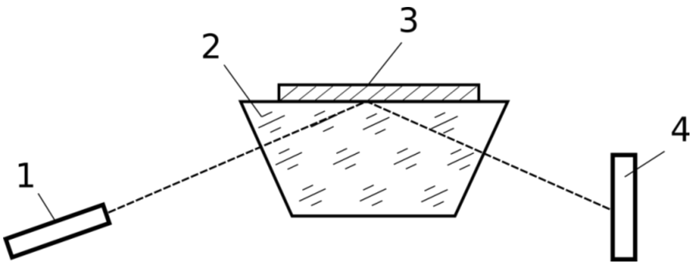

Fig. 2. Optical Scheme of the SPR Device Based on Kretschmann Geometry: 1 — laser, 2 — total internal reflection prism, 3 — sensor element, 4 — photodetector.

This compact device is suitable for autonomous rapid monitoring of pollution in natural ponds and rivers. This approach does not determine the specific composition of impurities but their total quantity by measuring the shift in the SPR minimum angle of the water samples being studied. The device's error at the research wavelength of 650 nm is 8×10^{-5} RIU. For repeated studies, a sensor element with an additional zinc oxide layer [25] was proposed, significantly enhancing the durability of the gold-sensitive layer. In these studies, the wear resistance of the gold layer was tested by performing multiple measurements on a single sensor with the additional layer, measuring the SPR minimum angle of various natural water samples.

Simultaneously with the SPR characteristics measurement, a conductometer was used to analyze water samples. We used TDS-3 conductometer (Chanitex, China) Fig. 3 to provide these experiments. Its technical description provided in Table 2.

Fig. 3. Appearance of conductometer TDS-3.

Table. 2. Technical description of conductometer TDS–3

Parameter	Value
Range, ppm	0–9.990
Accuracy, %	±2
Size, mm	155×31×23
Weight, g	76

For easier comparison of measurement results with the results obtained by the Ukrkhimanaliz laboratory, the measured conductivity was converted into ppm in accordance with the calibration curve provided by the manufacturer.

The objective of this study was to develop a methodology for rapid natural water quality assessment using the SPR method and a conductometer and to simultaneously determine the durability of sensors with protective coatings.

The first research stage involved modeling the concentration dependencies of SPR indicators by adding controlled amounts of organic (sugar) and inorganic (table salt and baking soda) impurities to distilled water. Biological contamination was modeled using suspensions of live yeast.

To investigate the influence of inorganic impurities on the SPR shift, we dissolved 1, 2.5, 3.5, 4.5, and 5.5 g of table salt (TM “1”) in 100 milliliters of distilled water using a magnetic stirrer.

To analyze the influence of organic impurities on the SPR shift, we dissolved 1.3, 2.7, 3.5, 4.5, and 5.5 g of sugar (TM “1”) in 100 milliliters of distilled water using a magnetic stirrer.

For studying the influence of pH on the SPR shift, we dissolved 0.7, 1.4, 2.1, 2.8, and 3.5 g of baking soda (TM “1”) in 100 milliliters of distilled water using a magnetic stirrer.

To model biological contamination, 20 g of pressed yeast (TM “Lvivski Premium”) were stirred in 300 milliliters of water at the room temperature (~22°C). The suspension was kept for 30 minutes at room temperature and measurements were carried out using conductometric and SPR methods. The obtained suspension was highly concentrated and exceeded the upper sensitivity limit of SPR measurements. Moreover, such a high concentration is not realistic in natural river water. After that, the suspension was kept for another 4 hours and repeated measurements were taken. Therefore, to better align with the research objectives, various amounts of water were added to the concentrated suspension, and a calibration SPR curve was constructed. Then, 1.3, 2.6, 4.0, 5.3, and 6.6 g of this suspension were mixed with 100 milliliters of distilled water for each sample.

For each sample, the angle of the SPR minimum was measured. The instrument's cuvette was cleaned with distilled water and ethanol after each measurement. Each measurement was repeated 10 times to estimate the measurement error. Additionally, we conducted 10 measurements of the conductivity of each sample. After each measurement, the conductometer was cleaned with distilled water.

In the second stage, four samples of coastal water were taken from different locations: the Dnipro River in Kyiv, the Stuhna River near Vasylkiv, a pond from the Stuhna River near Borova in the Fastiv district. Samples were collected between October 30 and November 4, 2024. Volume of each sample was 1 liter. For SPR and conductometry analyze samples of 50 ml volume were taken from each bottle. Rest volume of samples were transferred to the “Ukrkhimanaliz” during 12 hours. After each SPR measurement, the sensor element was rinsed with distilled water and alcohol to reduce errors from contamination by residues of previous samples. All measurements were carried out at room temperature (20 °C). However, during the measurements performed using the "Plasmon-6" device, the samples were heated by the instrument itself, which led to a decrease in the sample density, causing a shift of the SPR curve. So to minimize temperature influence we provide SPR measurements during 2 hours each sample.

To verify the accuracy of these rapid assessment methods, the water samples were additionally analyzed in the "Ukrkhimanaliz" laboratory using standard methods. Certificates of analyzed samples are stored in our laboratory.

Error of measurements was estimated as systematic error, which include influence of temperature, mechanical backlashes, etc., and random error. Systematic error for wavelength 650 nm is 8×10^{-5} RIU.

To determine the random measurement error, we used Student's method [26]. First, we calculated the arithmetic mean of the measured minimum SPR angle or conductivity using the formula:

$$a_m = \frac{\sum_{i=1}^{N} a_i}{N},$$

where a_m — the mean value of the measured quantity, a_i — the measured value, N — quantity of measurements.

After that, we determined the absolute deviation of the measured quantity from the mean:

$$\Delta a_i = |a_i - a_m|$$

And calculated the standard error of the mean [26]:

$$S = \sqrt{\frac{\sum_{i=1}^{N} \Delta a_i^2}{N(N-1)}},$$

where S — the standard error of the mean.

The confidence probability was taken as 0.95, and accordingly, the Student's coefficient for 10 measurements was 2.3 (for salt, sugar, yeast and conductometry measurements) and 1.98 for 1700 measurements. Thus, the value of the random error was calculated using the formula:

$$a_r = 2.3S$$

The minimum duration of analysis using the surface plasmon resonance (SPR) method is 1 minute, while the measurement with a conductometer takes 30–50 seconds. Thus, the total duration of the study does not exceed 2 minutes. The method allows for the assessment of the overall level of contamination and does not involve the identification of specific impurities. Preliminary studies were conducted on various substances to determine the sensitivity and selectivity of the conductometer. It was found that the conductometer is not sensitive to organic contaminants that do not conduct electric current (e.g., yeast suspension), which are present in river water. Therefore, to ensure objective monitoring, it was proposed to use two devices based on different physical principles.

RESULTS AND DISCUSSION

The research results are shown in Fig. 4–7.

The modeling conducted showed that for sensors with different sensitivities and designs, the concentration dependencies exhibit a common trend: a higher SPR minimum angle corresponds to a greater quantity of impurities. Thus, in monitoring natural water, the pollution level can be evaluated based on this parameter. More polluted water will have a larger SPR minimum angle.

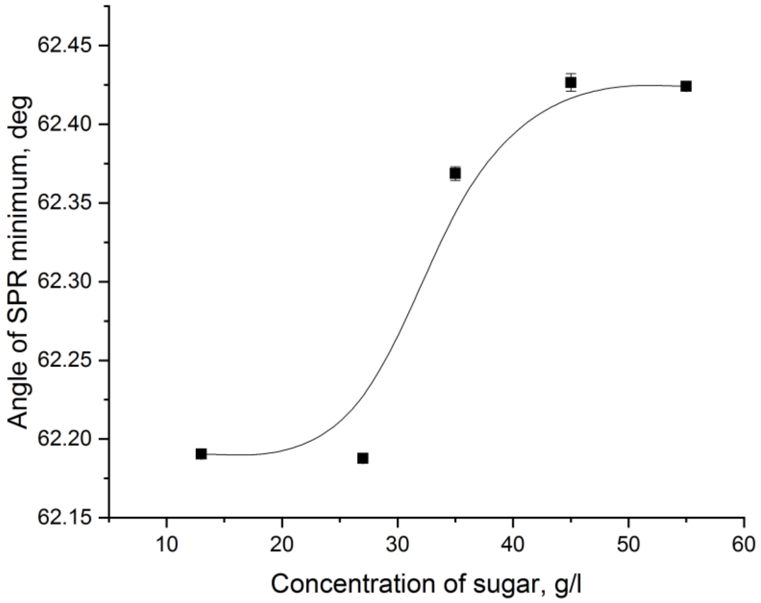

Fig. 4. Concentration dependence of the SPR minimum angle for sugar.

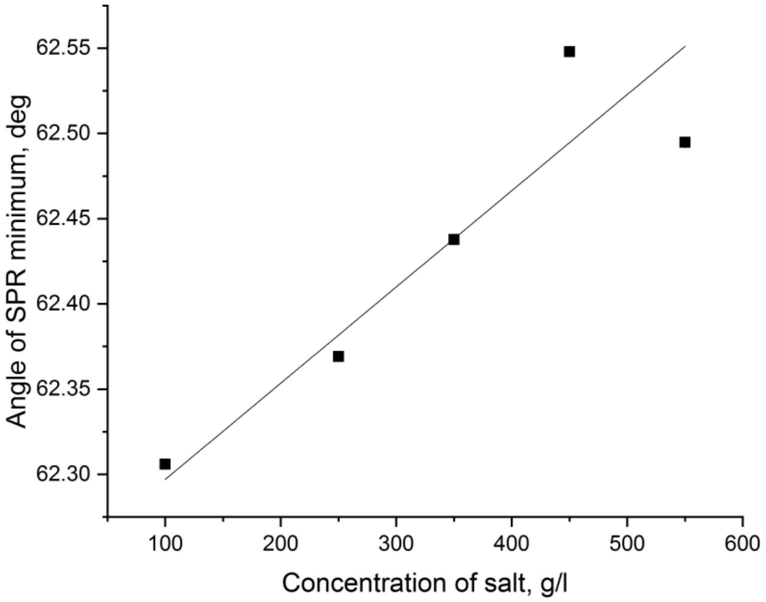

Fig. 5. Concentration dependence of the SPR minimum angle for salt.

In addition to the SPR minimum angle, samples were also analyzed using the conductometric method (Fig. 8).

After 30 minutes of preparation, the suspension showed zero conductivity on the conductometer.

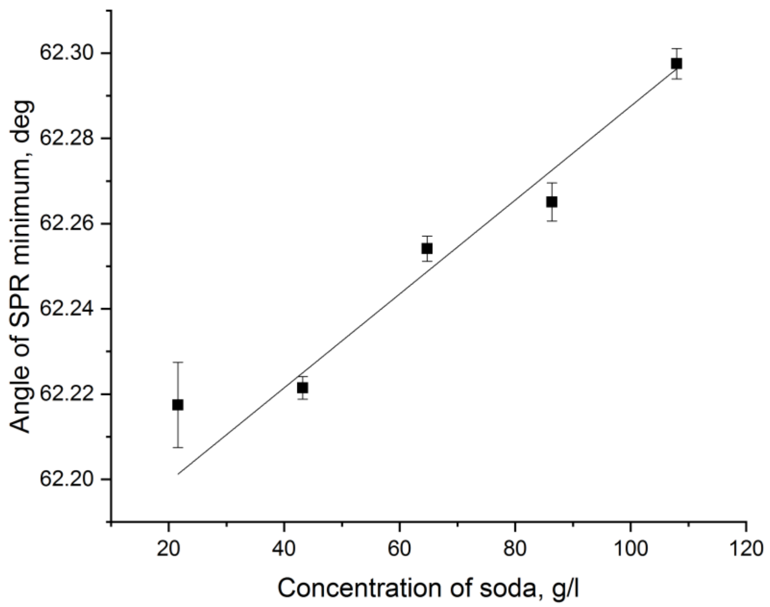

Fig. 6. Concentration dependence of the SPR minimum angle for soda.

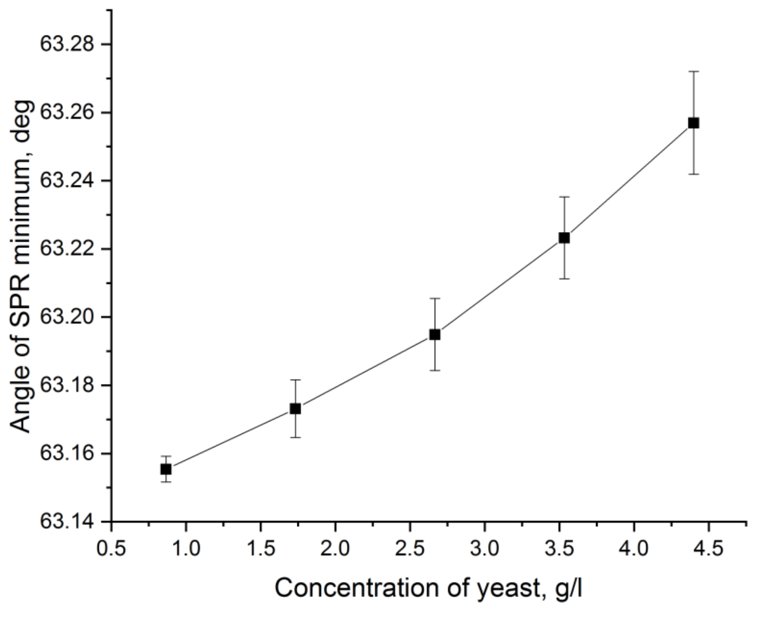

Fig. 7. Concentration dependence of the SPR minimum for aqueous yeast suspensions.

However, for organic impurities like sugar, this method was not informative, although it demonstrated a linear relationship for inorganic impurities. In measurements of yeast at a concentration of 20 g of "Lvivski Premium" pressed yeast per 300 ml of water, the following observations were made: 30 minutes after preparation, the suspension showed zero conductivity, but after 4 hours of activity, as a result of yeast activity, both organic and inorganic contaminants were formed in the suspension. The latter were detected by the conductometer as the conductive component. This result can be explained by the yeast's metabolic activity, which produces new chemical compounds, including acetic acid, known for its good conductivity.

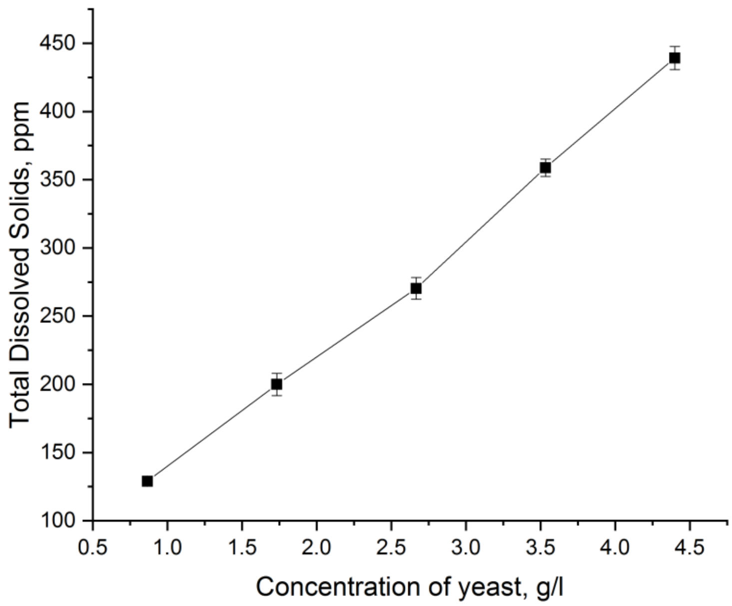

Fig. 8. Total dissolved solids for yeast after 4 hours of yeast's activity.

Consequently, it was decided to monitor water quality using both methods to ensure an objective assessment of natural water containing both organic and inorganic pollutants.

In Table 3 and Figs. 9, 10, the values of the SPR index and water conductivity are presented.

Table 3. Measurement results and comparison of selected parameters from laboratory analyses by "Ukrkhimanaliz" (from certificates)

Samples/ Measurement Results	Total Salt Content* mg/dm^3	Ammo-nium Concentration*, mg/dm³	Total Impurities, mg/dm^3	pH*	Adjusted Total Salt Content Considering Sample pH	SPR Degrees	Total Dissolved Solids, ppm
Dnipro	345	0.42	345.42	8	43.1775	62.2403± 5.49×10^{-4}	118.5±0.4
Stuhna	469	1.48	470.48	8	58.81	62.249± 1.57×10^{-3}	136.2±0.6
Pond	617	1.4	618.4	8	77.3	62.9619± 2.41×10^{-4}	144.6±0.5

Note: *Based on measurement results from "Ukrkhimanaliz".
**The instrumental error of the "Plasmon-6" SPR device at a wavelength of 650 nm is 8 × 10^{-5} RIU.

The measurement results can be summarized by stating that the combination of the surface plasmon resonance (SPR) method and conductivity measurement allows for the rapid, not exceed 2 minutes**,** and objective characterization of natural water pollution, which corresponds to the sum of selected harmful impurities. Given the small dimensions and autonomy of the devices used in the developed methodology, river water monitoring can be carried out in field conditions by one person.

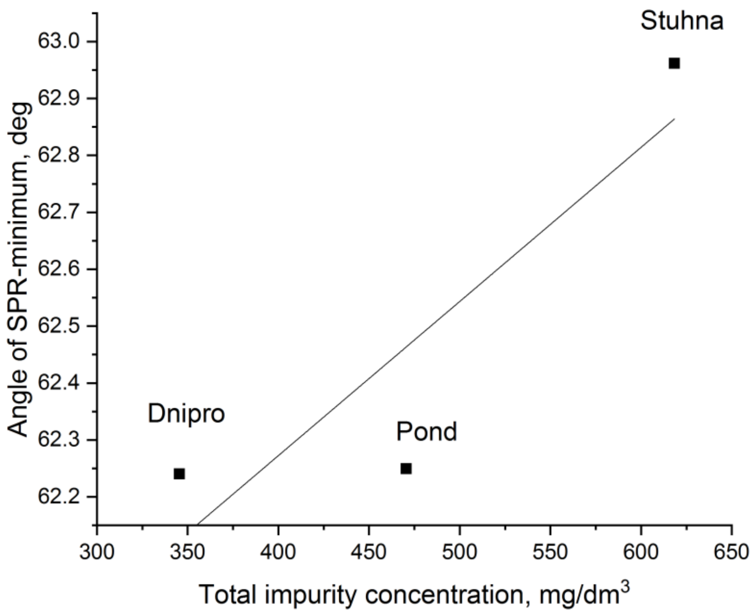

Fig. 9. Dependence of the SPR minimum angle on the total concentration of selected impurities in surface natural water samples.

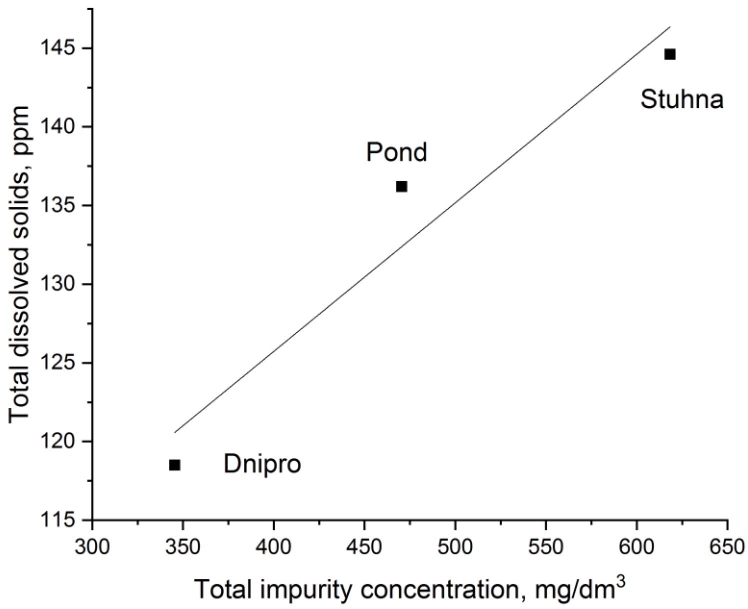

Fig. 10. Dependence of total dissolved solids on the total concentration of selected impurities in surface natural water samples.

All SPR measurements were carried out on a single sensor with an additional protective layer [26], which helped reduce the measurement error typically associated with sensor changes.

The SPR results showed that the most polluted river was Stuhna, followed by the pond, with the least pollution observed in the Dnipro River near Kyiv.

CONCLUSIONS

The measurement results can be summarized by stating that the combination of the surface plasmon resonance (SPR) method and conductivity measurement allows for the rapid, not exceed 2 minutes, and objective characterization of natural water pollution, which corresponds to the sum of selected harmful impurities. Given the small dimensions and autonomy of the devices used in the developed methodology, river water monitoring can be carried out in field conditions by one person.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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ЕКСПРЕС-МЕТОДИКА ДОСЛІДЖЕННЯ ЯКОСТІ ПРИРОДНОЇ ВОДИ ЗА ДОПОМОГОЮ СЕНСОРНОГО ПРИЛАДУ НА ОСНОВІ ПОВЕРХНЕВОГО ПЛАЗМОНОГО РЕЗОНАНСУ ТА КОНДУКТОМЕТРА

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Актуальність: Однією з актуальних проблем сучасності є забруднення природних вод, яке безпосередньо впливає на життєзабезпечення людства. Ця проблема пов'язана з інтенсифікацією

промисловості, сільського господарства та зміною клімату. Норми якості води в Україні визначаються державними стандартами, які регламентують як органолептичні показники, такі як каламутність і запах, так і допустимі концентрації шкідливих речовин.
Мета роботи: Метою даної роботи була розробка нового високочутливого методу визначення якості води.
Матеріали і методи: У цьому дослідженні визначається доцільність поєднання поверхневого плазмонного резонансу (ППР) і кондуктометричного методів для моніторингу якості природної води. Перший етап включав моделювання концентраційних залежностей параметрів ППР та електропровідності при додаванні до дистильованої води контрольованих кількостей органічних (цукор) і неорганічних (кухонна сіль і сода) домішок. Біологічне забруднення моделювали за допомогою розчинів живих дріжджів. Згодом було проаналізовано проби прибережної води р. Дніпро в Києві, р. Стугна біля м. Василькова та ставка, що сполучається з р. Стугна біля с. Борова Фастівського району. Усі ППР-дослідження проводилися з використанням покращеного сенсорного елемента з додатковим захисним шаром оксиду цинку, який зменшив помилки вимірювання, зазвичай пов’язані із заміною датчика. Для перевірки достовірності методів експрес-оцінки зразки води додатково досліджено стандартними лабораторними методами в компанії ТОВ «Укрхіманаліз».
Результати: Результати ППР-досліджень показали, що найбільш забруднена р. Стугна, за нею ставок, а найменше забруднення у р. Дніпро.
Висновки: Підсумовуючи результати вимірювань можна зробити висновок, що поєднання ППР-вимірювань і електропровідності дозволяє швидко та об’єктивно оцінити рівень забруднення природних вод. Це відповідає загальній кількості шкідливих домішок, нормованих на одиницю значення pH. Повторні дослідження з використанням вдосконаленого сенсорного елемента підтвердили, що оксид цинку як додатковий шар на сенсорі ефективно захищає золоте нанопокриття, зменшує помилки, пов’язані із заміною сенсора, і значно продовжує термін експлуатації сенсора, зберігаючи низький рівень помилок.
КЛЮЧОВІ СЛОВА: поверхневий плазмонний резонанс; електропровідність; контроль природної води; забруднення природних вод; хімічний аналіз; оптичні властивості.