



# TARGET COMPREHENSIVE PROGRAM OF SCIENTIFIC RESEARCH OF THE NAS OF UKRAINE

*Development of scientific principles of hydrogen  
production, storage and use in autonomous energy  
supply systems*



**Portable photoelectrochemical cells with hydrogen  
accumulation**



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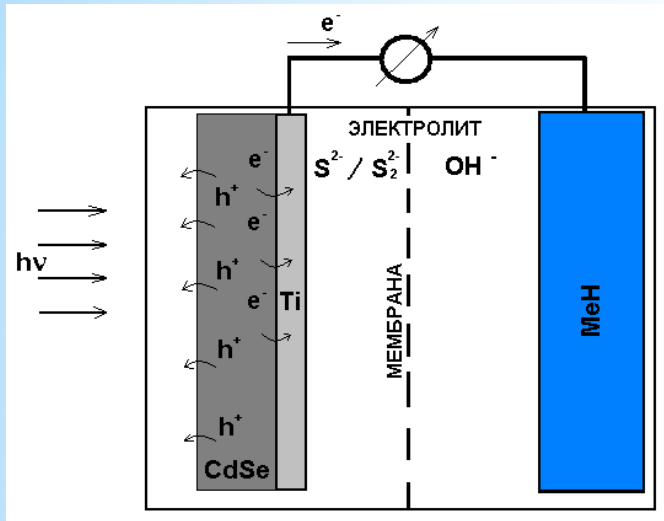
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# Basic photoelectrochemical cell for generating and hydrogen accumulation



## The advantages of this cell:

- optimization of electrochemical processes occurring in both parts of the cell
- the use of narrow-gap semiconductor photoanodes that absorb visible light
- accumulation of hydrogen directly at the cathode

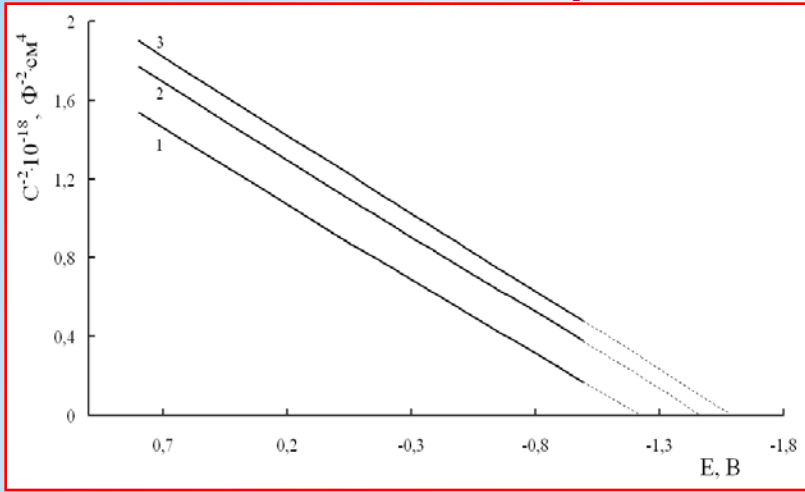


The aim of the project is to develop photoelectrochemical cells to convert sunlight energy into hydrogen energy with sufficient efficiency from aqueous electrolyte solutions and hydrogen accumulation directly in the photoelectrochemical cell at the cathode which consist from hydrogen-absorbing alloy. Development of alternative designs in which, in the absence of lighting, the chemical energy of hydrogen accumulated at the cathode will be converted into electrical energy.

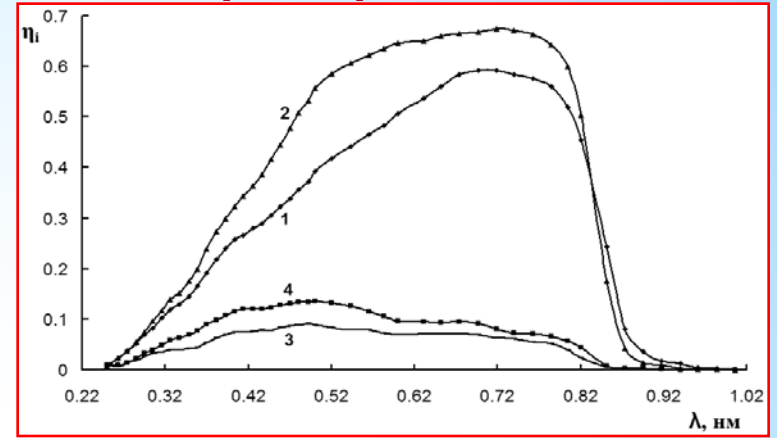
### **The problems that were solved within the framework of the Program:**

- Development new photosensitive semiconductor heterostructures and nanocomposites with a wide spectrum of sunlight absorption;**
- Use the effective electrode materials for hydrogen evolution and its accumulation and matching the characteristics of the photoanode and cathode;**
- Selection of electrochemical systems to minimize losses in the cell due to the influence of the electrolyte properties, as well as prevent photocorrosion and stabilize the electrolyte consist;**
- Search for alternative electrochemical systems and designs to improve the performance of the photoelectrochemical cell.**

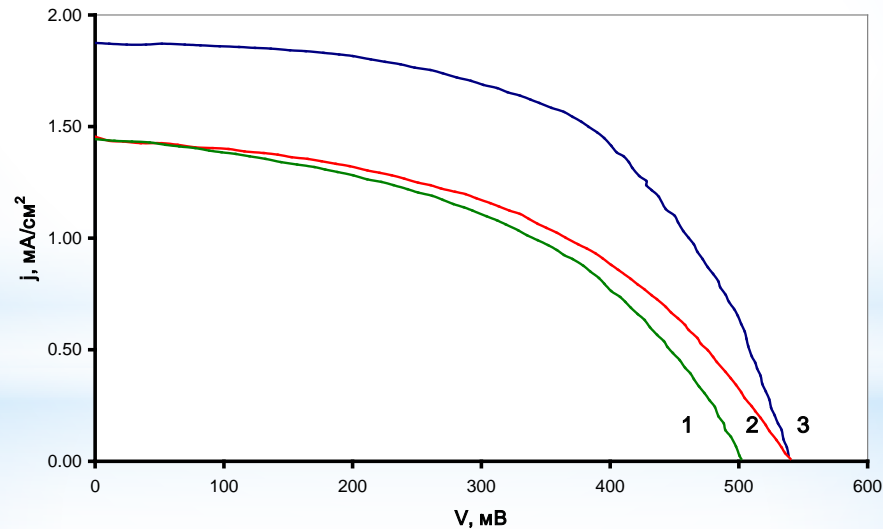
# Photocurrent spectra and the value of the photopotential



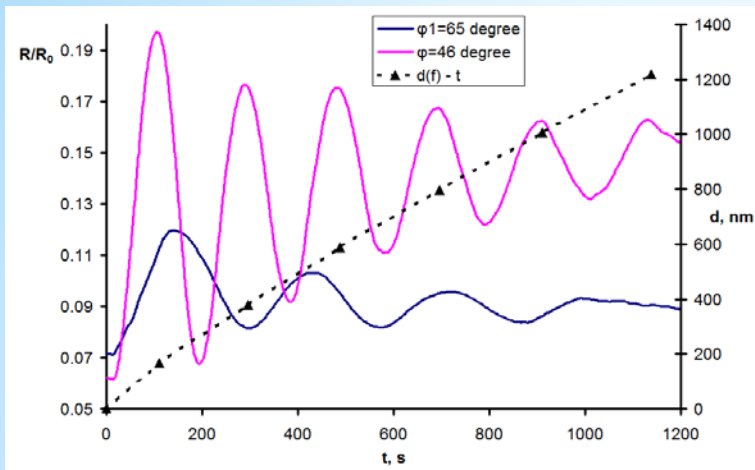
Potential of flat bands CdSe - electrode, modified by quantum points Zn (2), Pt (3), (1) – initial sample



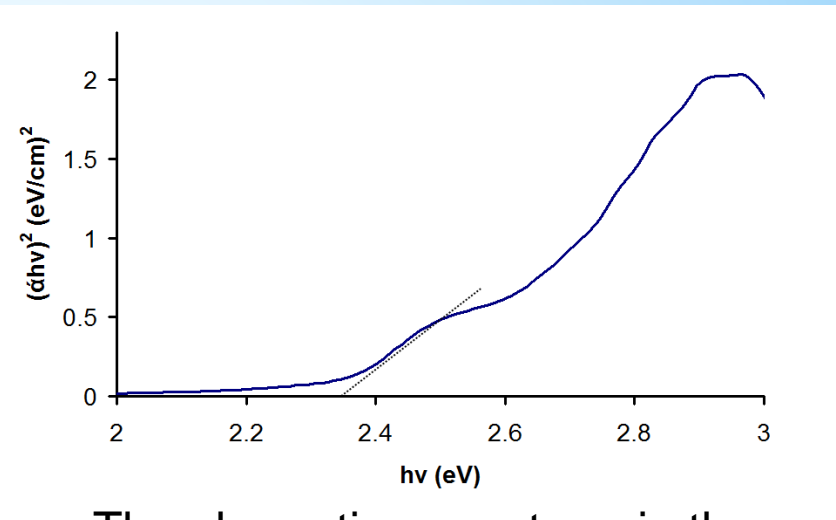
Spectral dependence of the quantum yield of the photocurrent  $\eta_i$  in a 1N solution NaOH for the initial CdSe<sub>0.65</sub>Te<sub>0.35</sub> electrode (3,4) and for the modified ZnS (1) and RGO (2).



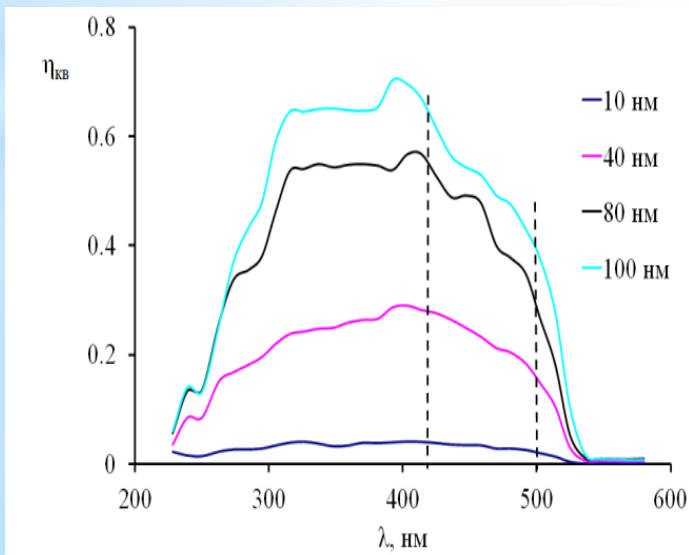
Volt-ampere characteristics of polycrystalline CdSe electrodes in polysulfide solution (containing 1M KOH, Na<sub>2</sub>S, S), P = 16 mW / cm<sup>2</sup>. 1 - after etching in HCl: HNO<sub>3</sub>; 2 - after etching in CrO<sub>3</sub> or K<sub>2</sub>CrO<sub>4</sub>; 3 - after photodigestion and treatment by K<sub>2</sub>CrO<sub>4</sub>.



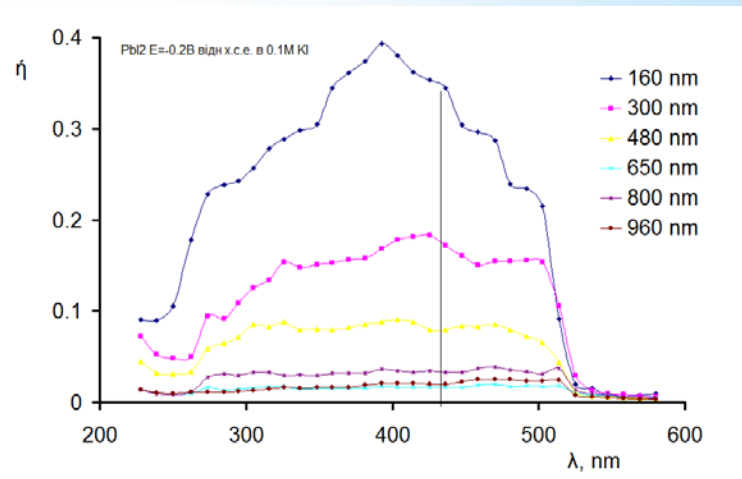
Interference pattern of the electrodeposition process  $\text{SnO}_2 / \text{PbO}_2$



The absorption spectrum in the coordinates  $(\alpha h\nu)^2$  from  $h\nu$  (eV).

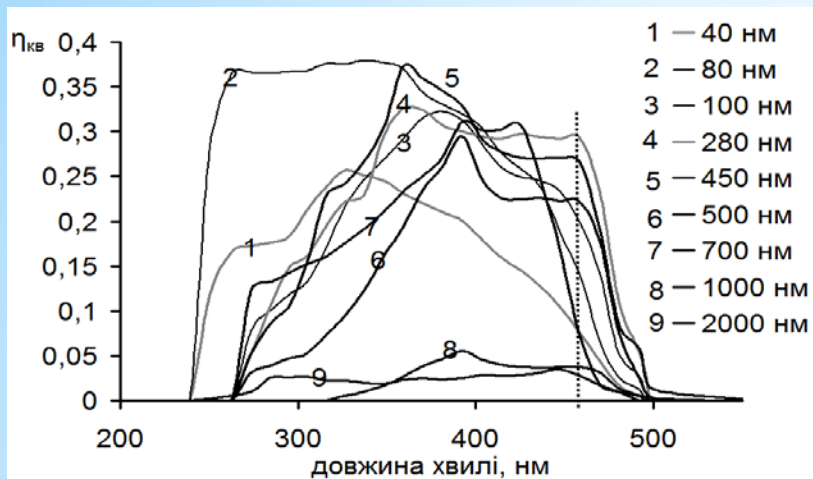


$\text{SnO}_2 / \text{PbI}_2$  quantum yield of thin films

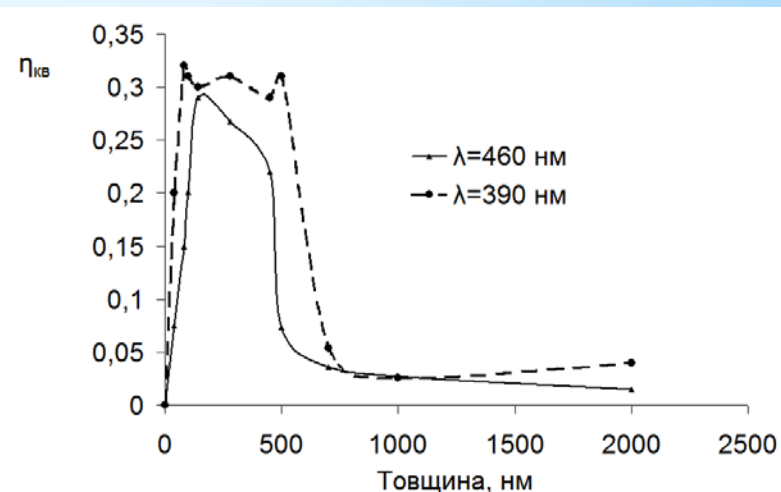


$\text{SnO}_2 / \text{PbI}_2$  quantum yield of thick films

## Quantum yield of SnO<sub>2</sub> / BiVO<sub>4</sub> films

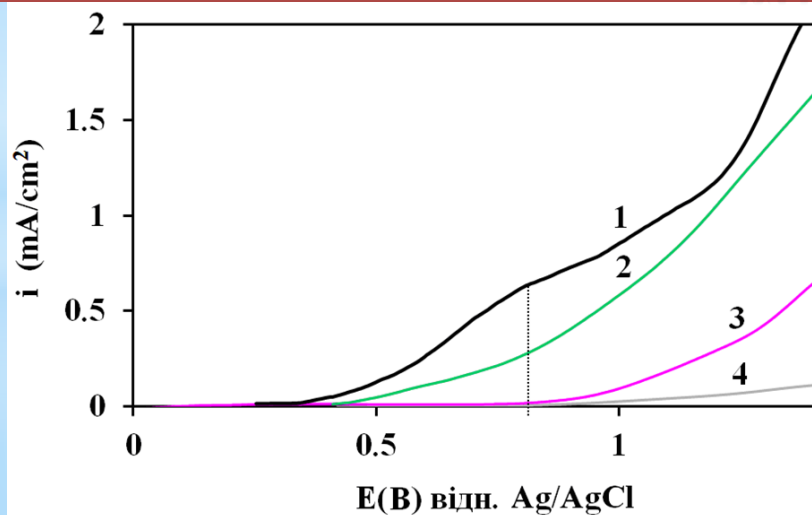


Spectra of quantum yield of photocurrent of SnO<sub>2</sub> / BiVO<sub>4</sub> films with different thickness

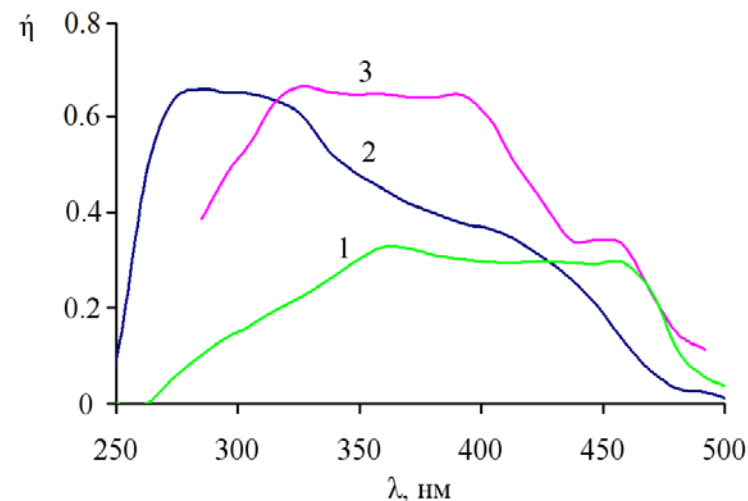


Dependence of the quantum yield of the photocurrent of SnO<sub>2</sub> / BiVO<sub>4</sub> films of the thickness by light illumination  $\lambda = 460$  nm and  $\lambda = 390$  nm

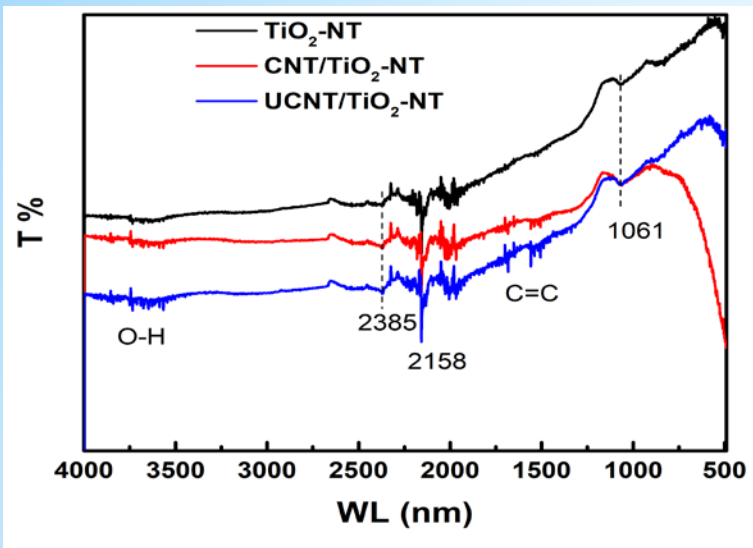
## Volt-ampere characteristics and spectral dependences of the quantum yield of the photocurrent of WO<sub>3</sub> / BiVO<sub>4</sub> films



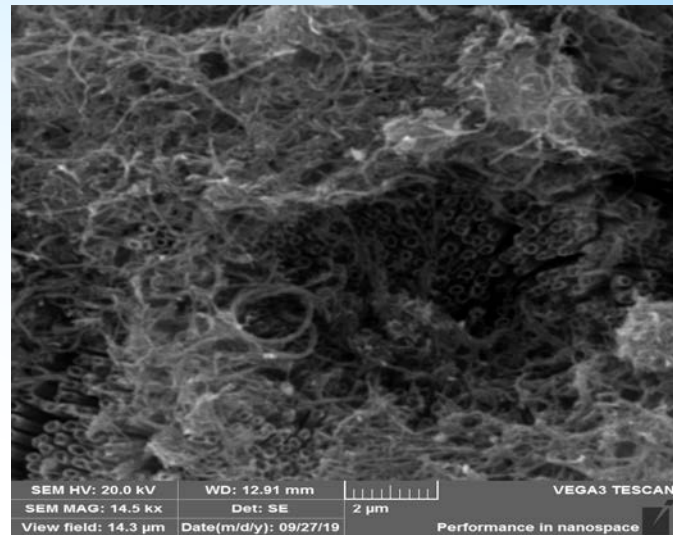
Volt-ampere characteristics of films when illuminated by a fluorescent lamp (illumination power 100 mW / cm<sup>2</sup>): WO<sub>3</sub> / BiVO<sub>4</sub> (500/140 nm) (1), BiVO<sub>4</sub> (140 nm) (2), BiVO<sub>4</sub> (700 nm) (3), dark current (4).



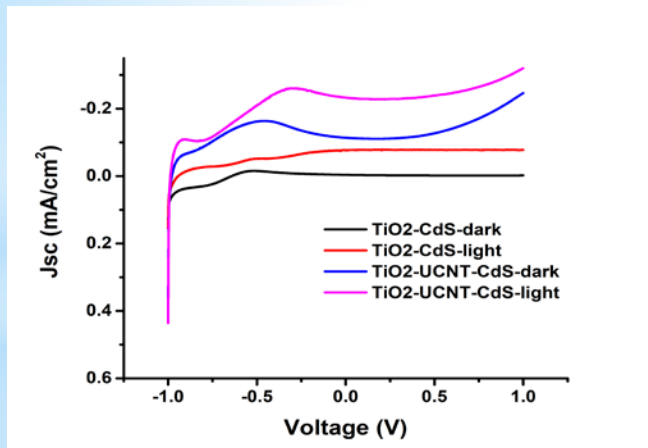
Spectral dependences of the quantum yield of photocurrent for 1 - BiVO<sub>4</sub> film, 2 - heterostructure and 3 - BiVO<sub>4</sub> composite with WO<sub>3</sub> in a solution of 0.1 M Na<sub>2</sub>SO<sub>4</sub> at a potential of +0.8 V vs SCE .



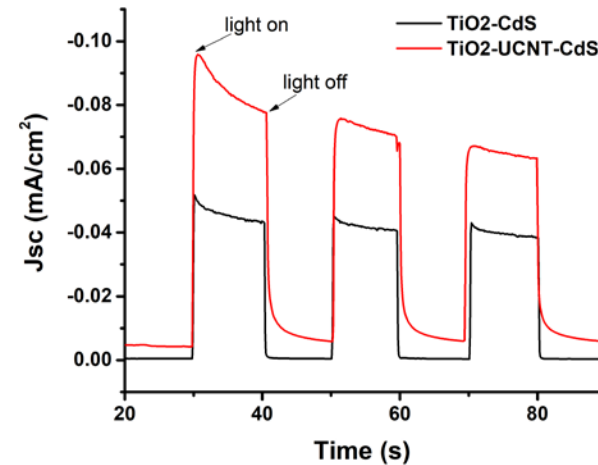
IR spectra: for the initial sample of NT-TiO<sub>2</sub>; modified by carbon nanotubes NT-TiO<sub>2</sub> / CNT; modified by partially unzipped (UCNT) NT-TiO<sub>2</sub> / UCNT carbon nanotubes.



Electron micrograph heterostructure of NT-TiO<sub>2</sub> / CdS modified by partially unzipped nanotubes

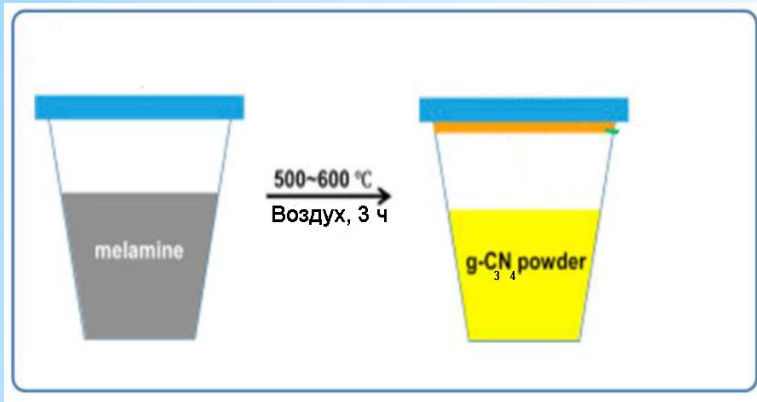


Current-voltage characteristics in the dark and at the illumination for heterostructure of the NT-TiO<sub>2</sub> / CdS and modified by partially unzipped carbon nanotubes (NT-TiO<sub>2</sub> / UCNT / CdS). P = 16 mW / cm<sup>2</sup>

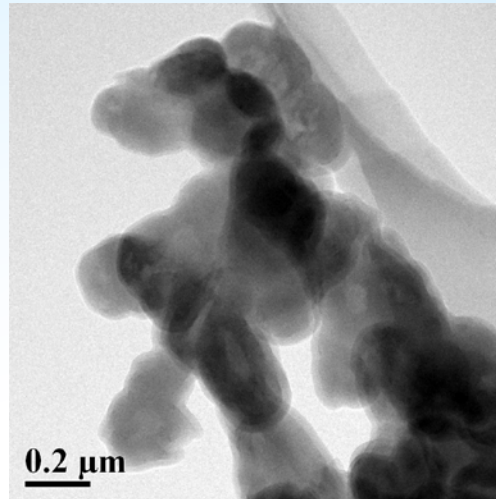


Relaxation of the photocurrent at illumination for the NT-TiO<sub>2</sub> / CdS heterostructure and modified by partially unzipped carbon nanotubes (NT-TiO<sub>2</sub> / UCNT / CdS). P = 16 mW / cm<sup>2</sup>

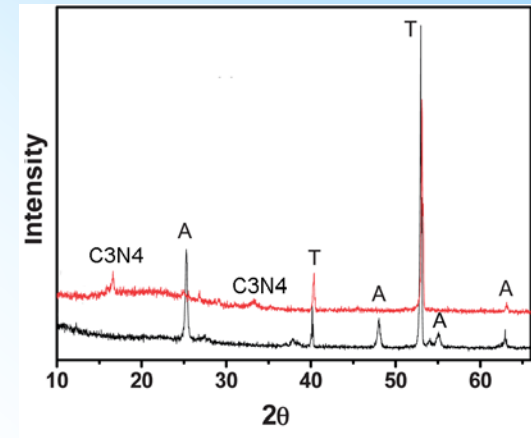
# Photosensitive nanocomposites $\text{TiO}_2 / \text{C}_3\text{N}_4$



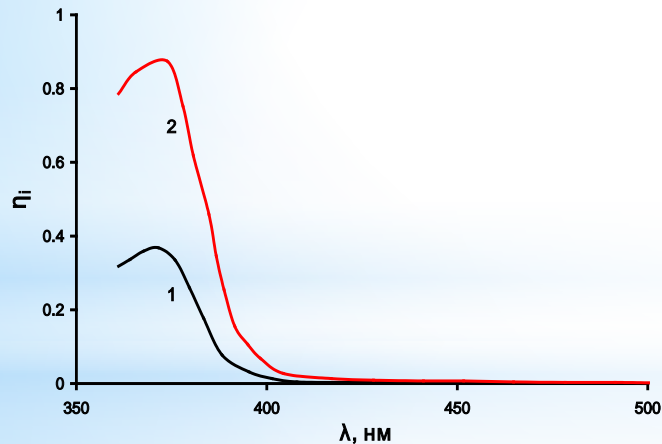
Schematic illustration of  $\text{C}_3\text{N}_4$  production.



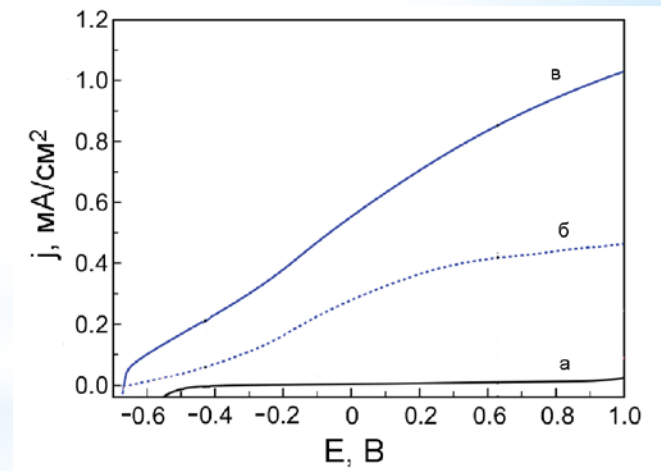
Electronic photomicrograph of the  $\text{g-C}_3\text{N}_4$  sample



XRD of  $\text{TiO}_2$  nanotubes (a) and  $\text{TiO}_2 / \text{C}_3\text{N}_4$  nanocomposite (b).

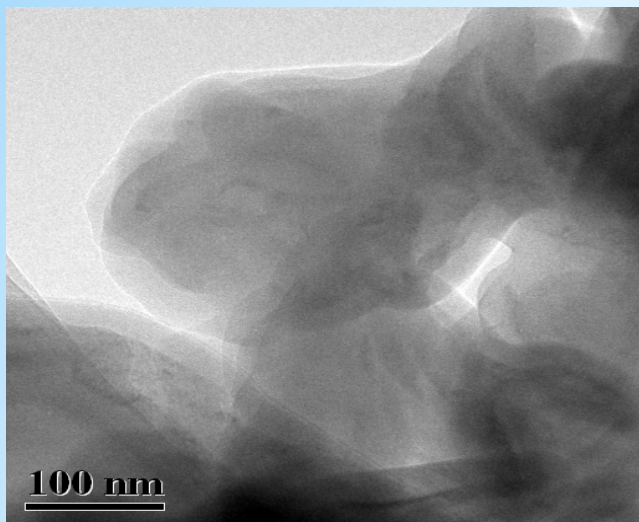


Quantum yield spectra of photocurrent on  $\text{TiO}_2$  (1) and  $\text{TiO}_2 / \text{C}_3\text{N}_4$  (2) deposited from a suspension of  $\text{C}_3\text{N}_4$  in methanol after 20 minutes of sonication. Potential - +0.6 V vs. s.c.e.

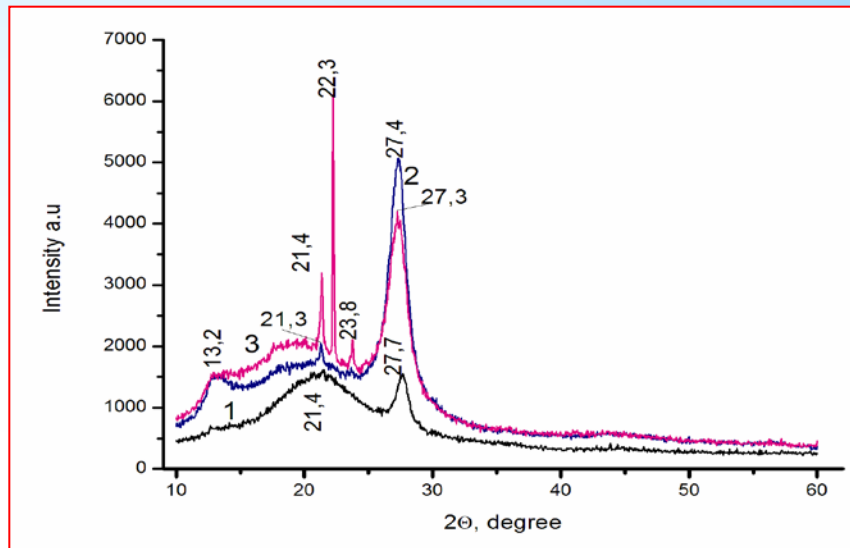


Dependence of photocurrent density on the potential of  $\text{TiO}_2$  nanotubes under light (b), in the dark (a) and  $\text{TiO}_2 / \text{C}_3\text{N}_4$  nanocomposite (c). Lighting power 50  $\text{mW} / \text{cm}^2$ .

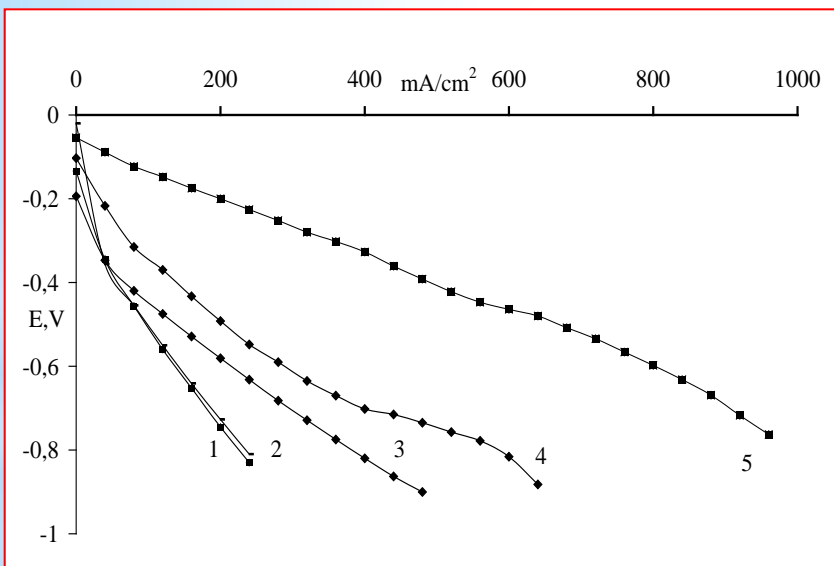




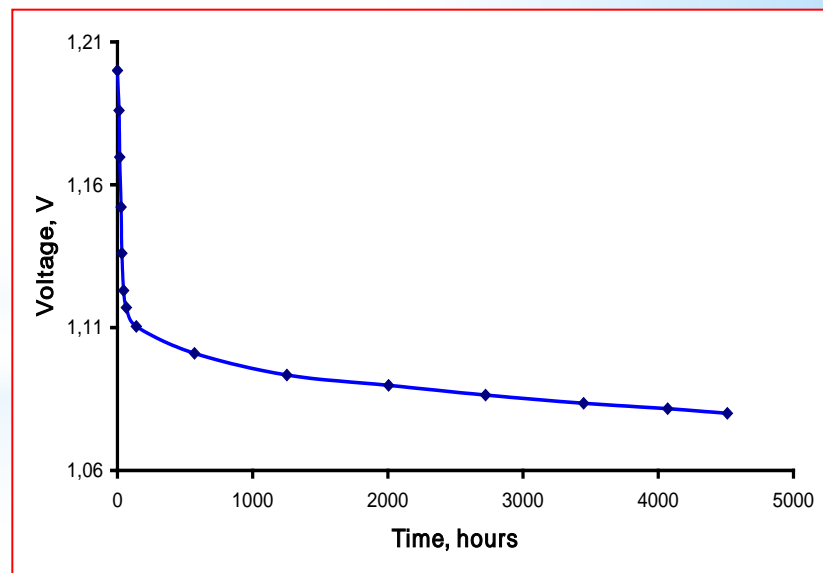
Electron micrograph of sample g-C<sub>3</sub>N<sub>4</sub> (2: 1).



XRD patterns for carbon nitride with different ratios of urea to melamine in the synthesis: 56.5: 1 (1); 1: 1 (2); 2: 1 (3)

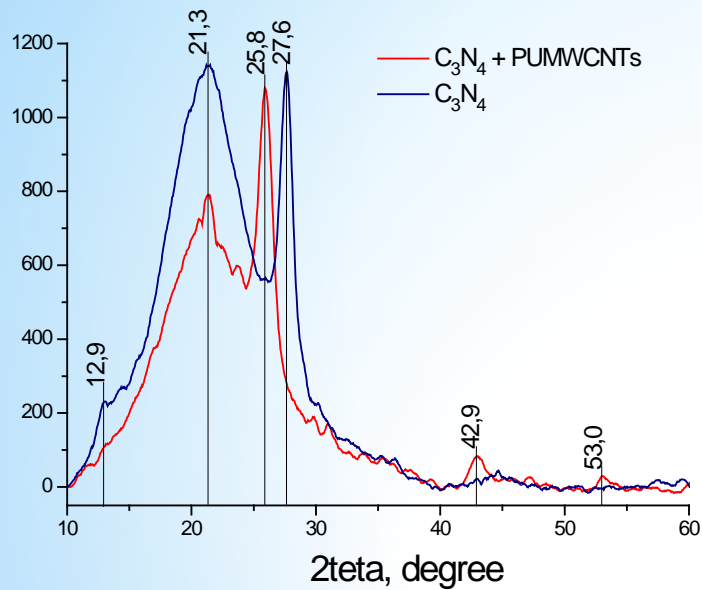


Current-voltage characteristics of oxygen electrodes with an active layer of various electrode materials: 1 - g C<sub>3</sub>N<sub>4</sub> (1: 1) - melamine on top; 2 - g C<sub>3</sub>N<sub>4</sub> (1: 1) the mixture is stirred; 3 - g C<sub>3</sub>N<sub>4</sub> (56.5: 1); 4 - g C<sub>3</sub>N<sub>4</sub> (2: 1); 5 - MWCNT with deposited platinum in the amount of 10 wt.%.



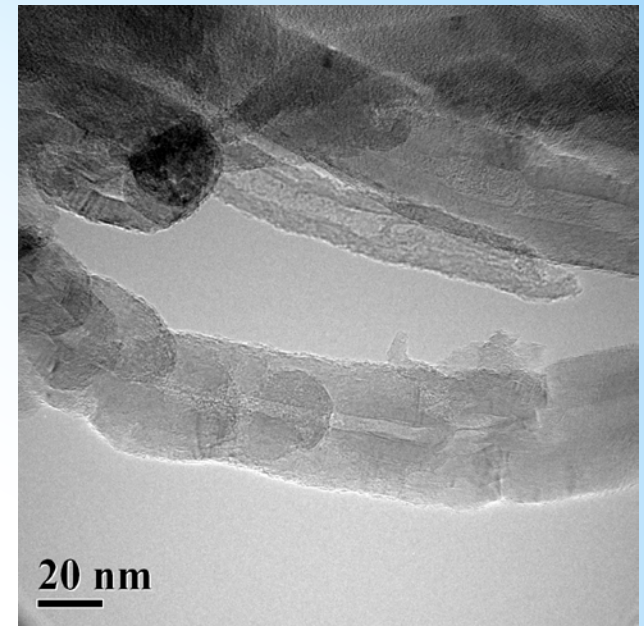
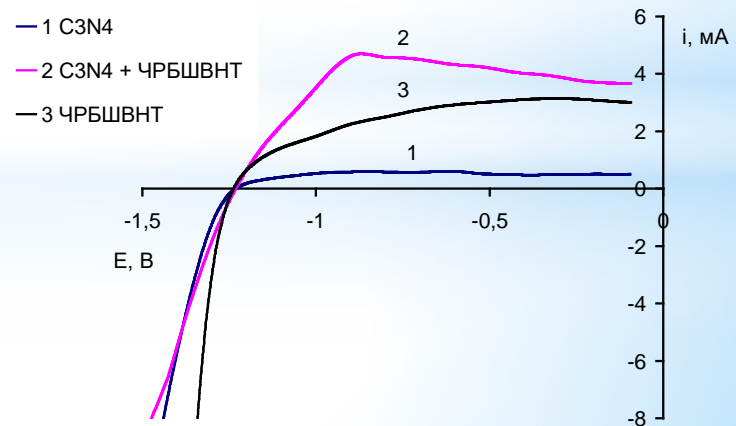
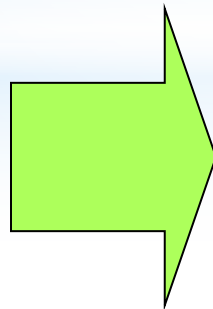
Results of long-term tests of oxygen electrode with an active layer consisting of carbon nitride at constant current density of 200 mA cm<sup>-2</sup>.

# Hybrid composite - partially unzipped carbon nanotubes / $C_3N_4$



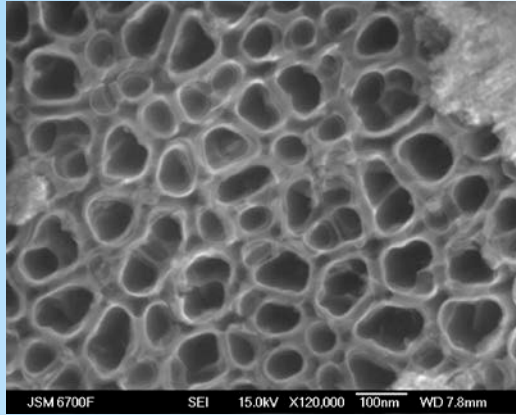
X-ray diffraction of a hybrid nanocomposite of layered structures of graphitized carbon nitride and partially unzipped multiwalled carbon nanotubes

Cyclic volt-ampere curves for hydrogen evolution for electrodes of partially unzipped carbon nanotubes (3), graphit like carbon nitride (1) and hybrid nanocomposite of layered structures of graphit like carbon nitride and partially unzipped multiwalled carbon nanotubes (2)

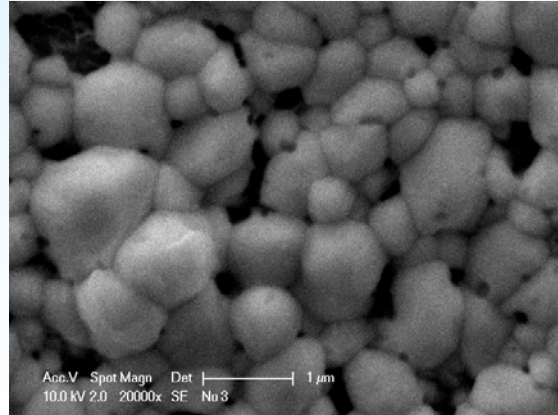


Photomicrograph of a hybrid nanocomposite of layered structures of graphitized carbon nitride and partially unzipped multiwalled carbon nanotubes.

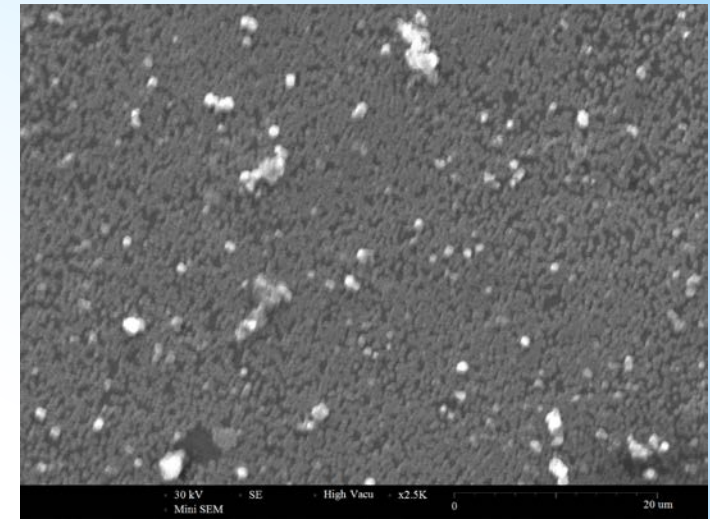
# ELECTRODES WITH A CdSe / ZnSe HETEROJUNCTION



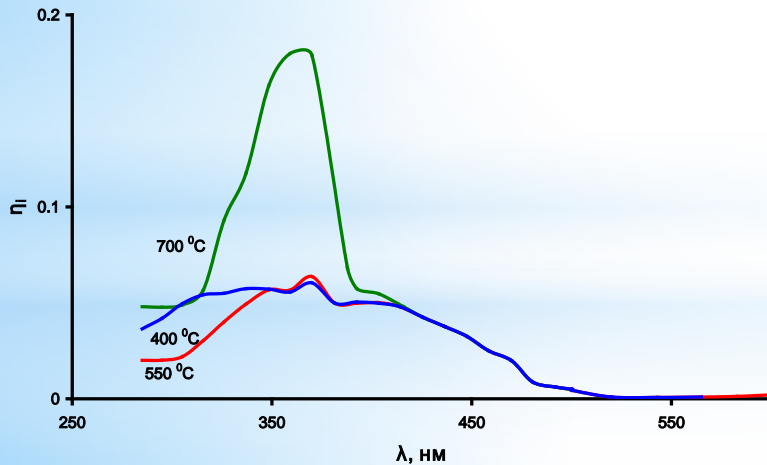
Electronic micrograph of TiO<sub>2</sub> nanotubes.



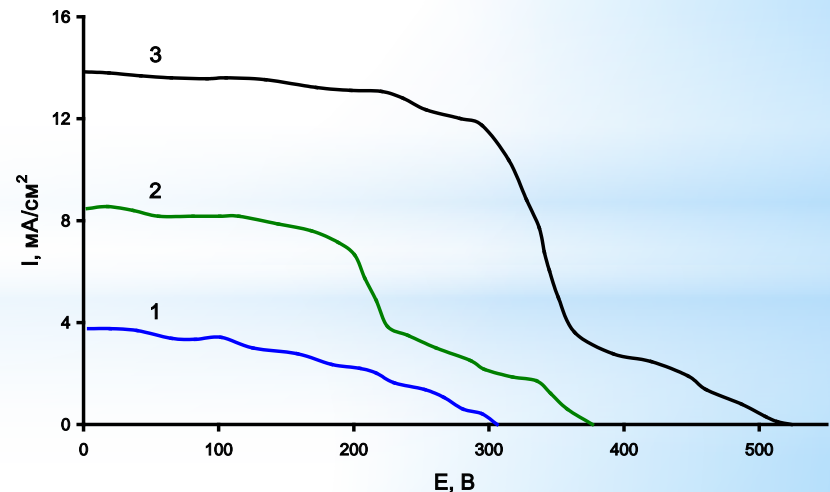
Micrograph of NT-TiO<sub>2</sub> / CdSe film.



Micrograph of hydrochemically precipitated ZnSe film annealed at 700 °C.

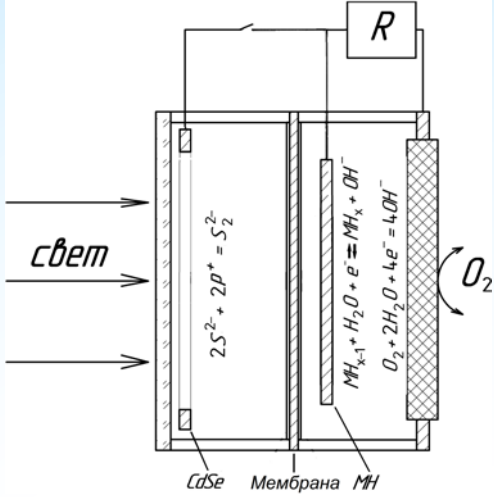


Quantum yield of zinc selenide photocurrent at different annealing temperatures.



Volt-ampere curves of photoelectrochemical systems made of single-layer semiconductors ZnSe (1), CdSe (2) and the CdSe / ZnSe heterosystems(3). Cathode - Cu<sub>2</sub>S .

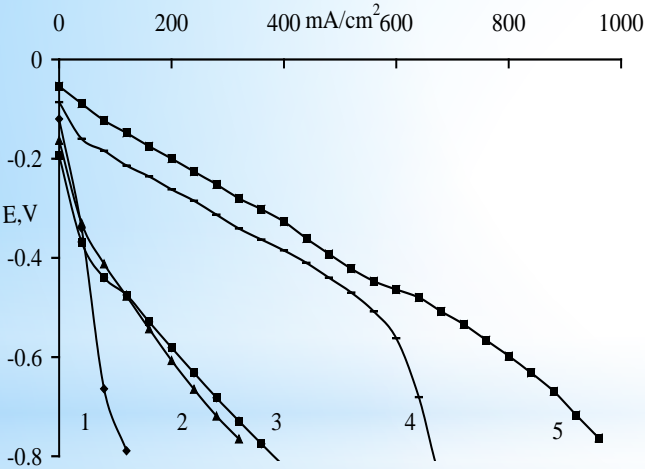
# COMPOSITE ELECTRODE MATERIALS FOR A SOLAR CELL WITH HYDROGEN ACCUMULATION



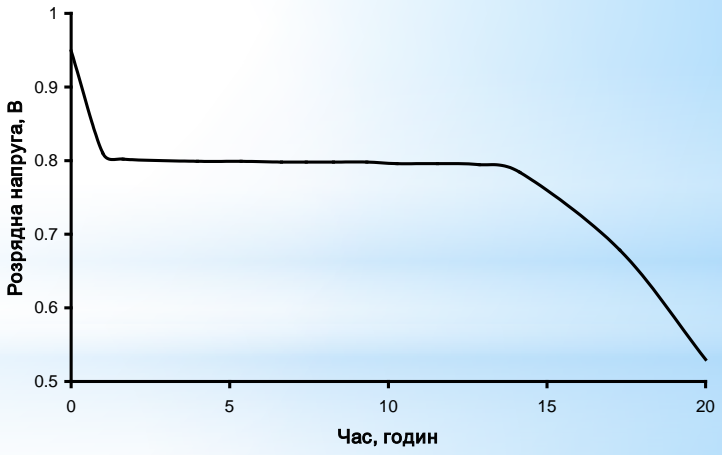
Scheme of electrochemical fuel cell with photoelectrode and MH / air electrode system.



Electrochemical fuel cell with photoelectrode and MH / air electrode system.



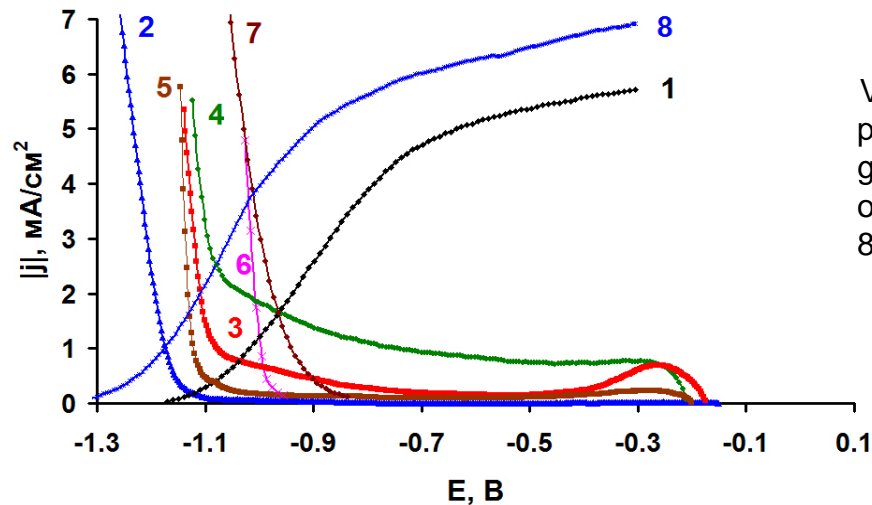
Dependence of potential (E) on current density (j) for oxygen electrodes with an active layer in the amount of 0.02 g / cm<sup>2</sup>, consisting of: 1 - initial multiwalled carbon nanotubes; 2 - partially unzipped by electrochemical method by 2 hours oxidation multiwalled carbon nanotubes in sulfuric acid; 3 - thermally synthesized carbon nitride (g-C<sub>3</sub>N<sub>4</sub>); 4 - hybrid nanocomposite of layered structures of graphitized carbon nitride and partially unzipped multiwalled carbon nanotubes; 5 - multiwalled carbon nanotubes coated by Pt.



Change of voltage on a cell at the discharge current of 20 mA in 7 n KOH solution with the use the air electrode

# MH electrode : $\text{LaNi}_{2.5}\text{Co}_{2.4}\text{Al}_{0.1}$

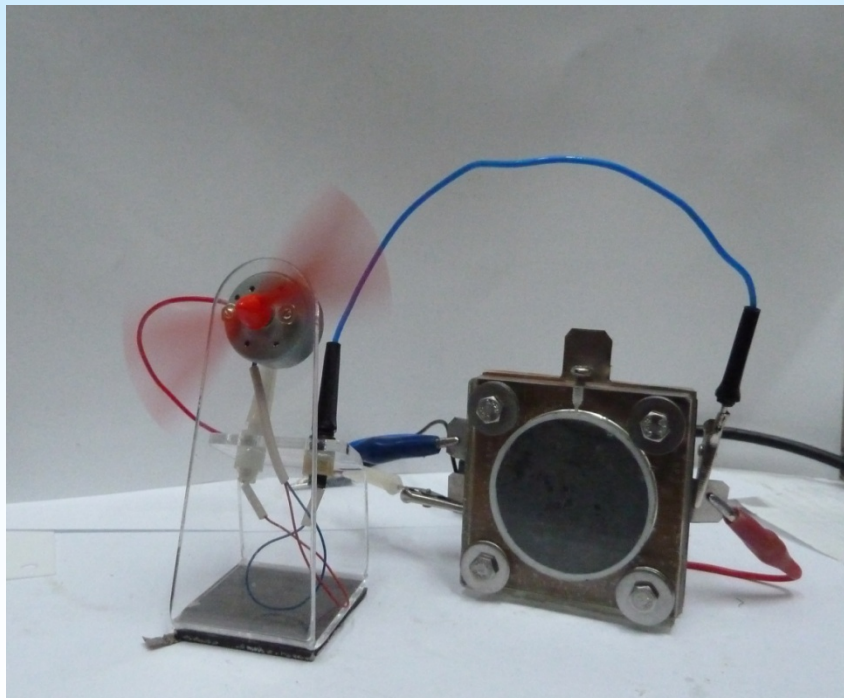
Discharge capacity of the electrode  $175 \div 258 \text{ mAh/g}$



Volt-ampere characteristics of the electrodes of the photoelectrochemical cell: 1- CdSe photoanode; 2 - graphene oxide; 3 - RGO; 4 - RGO modified with Cr oxide; 5 - Pt on nickel; 6 -  $\text{LaNi}_4\text{Mn}_{0.5}\text{Al}_{0.5}$ ; 7 -  $\text{LaNi}_3\text{Co}_2$ ; 8 - CdSe/ZnSe + RGO.

Table. The value of the potential of open circuit for CdSe electrodes without illumination and characteristics of the photoelectrochemical cell at  $P = 16 \text{ mW / cm}^2$  for the solution containing 1M of  $\text{OH}^-$ ,  $\text{S}^{2-}$  and S depending of cationic composition at room temperature.

Cation	Potential $E_0$ (V vs SCE )	High-voltage (V)	Short circuit current ( $\text{mA/cm}^2$ )
$\text{Li}^+$	-0.719	0.450	0.92
$\text{Na}^+$	-0.727	0.542	1.73
$\text{K}^+$	-0.747	0.547	1.87



### Photoelectrochemical cell characteristics

Electrode		Photo-current, mA	Current density, mA / cm <sup>2</sup>		Potential E, V	
anode	cathode		anode	cathode	anode	cathode
CdSe/ZnSe +RGO	LaNi <sub>2.5</sub> Co <sub>2.4</sub> Al <sub>0.1</sub>	23.3	7.19	3.76	-	-0.924
CdSe+RGO		20.6	6.05	3.32	-0.964	-0.928
CdSe		16.5	5.44	2.66	-0.951	-0.918
CdSe+ZnS		18.5	6.31	2.98	-	-0.926

## CONCLUSIONS

1. There are formed photosensitive materials based on nanoheterostructures and nanocomposites of cadmium and zinc chalcogenides, their solid solutions, bismuth vanadates, graphene-like structures and metal oxides. It was found that the creation of nanoheterostructures and nanocomposites increases their photocatalytic activity, improves charge separation and inhibits recombination processes, which leads to an increase in the photopotential of electrodes based on these materials, as well as their efficiency in the photoelectrochemical cell to produce hydrogen.
2. Was obtained porous graphite like carbon nitride ( $g\text{-C}_3\text{N}_4$ ) by simple methods of pyrolysis of melamine and urea. Photosensitive nanocomposites  $\text{TiO}_2 / \text{C}_3\text{N}_4$  from the emulsion of methyl alcohol and  $\text{C}_3\text{N}_4$  were obtained. At the obtained heterojunction, of the graphene-like structure of  $g\text{-C}_3\text{N}_4 / \text{TiO}_2$  nanotubes enhances the light absorption in the visible part of the spectrum and at the same time accelerates the charge distribution between  $\text{TiO}_2$  and  $g\text{-C}_3\text{N}_4$  nanotubes and increases the load characteristics of the photoelectrochemical for hydrogen accumulation.
3. A hybrid nanocomposite was synthesized from layered structures of graphite like carbon nitride and partially unzipped multilayer carbon nanotubes. It is shown that the developed material is promising for use as an electrode material in a photoelectrochemical cell to obtain "solar hydrogen".
4. The developed techniques were used for the obtaining photosensitive electrode materials of photoelectrochemical systems for the production and accumulation of hydrogen. The optimal consist of electrolytes are determined and improved design the photoelectrochemical system with air and hydrogen-absorbing electrodes. From the discharge curves obtained on the MH-air cell, it was found that the voltage on the cell is  $\sim 0.8$  V at the discharge current of 20 mA in 7N KOH solution using an air electrode in the case when the MH cathode is pre-charged in pair with the photoanode. The obtained results shows to the prospects of the developed system for use in an electrochemical photoaccumulator.

# PUBLICATIONS OF THE AUTHORS OF THE 2019-2021 REPORT

## Articles

1. I.A. Rusetskiy, M.O. Danilov, S.S. Fomanyuk, I.A. Slobodyanyuk, V.S. Vorobets, G.Ya. Kolbasov. Photoelectrochemical properties of the composites based on TiO<sub>2</sub> nanotubes, CdSe and graphene oxide. // Research on Chemical Intermediates, – 2019. – V.45, №8 – P. 4121–4132. DOI:10.1007/s11164-019-03895-0
2. Smilyk V.O., Fomanyuk S.S., Kolbasov G.Ya., Rusetskiy I.A., Vorobets V.S. Electrodeposition, optical and photoelectrochemical properties of BiVO<sub>4</sub> and BiVO<sub>4</sub>/WO<sub>3</sub> films // Research on chemical intermediates, – 2019. –V.45, №8 – Т №45 – P. 4149–4161 <https://doi.org/10.1007/s11164-019-03897-y>
3. Л.Г. Щербакова, І.А. Русецький, Д.В. Патлун, М.О. Данилов, С.С. Фоманюк, В.О. Смілик, М.О. Лапа, В.С. Воробець, Г.Я. Колбасов, Ю.М. Солонін. Електрохімічна паливна комірка з фотоелектродом та системою МН / повітряний електрод. // Відновлювальна енергетика та енергоефективність у XXI столітті: матеріали ХХ міжнародної науково-практичної конференції (Редактори: Кудря С.О., Резцов В.Ф., Суржик Т.В., Репкін О.О., Кузнецов М.П., Васько П.Ф., Морозов Ю.П., Головка В.М., Будько В.І., Ключ В.П., Ключ С.В., Пепелов О.В.) (Київ, 15-16 травня 2019 р.).- К.: Інтерсервіс, 2019, с. 181-185.
4. M.O. Danilov, I.A. Rusetskii, I.A. Slobodyanyuk, G.I. Dovbeshko, O.Y. Khyzhun, V.V. Strelchuk, G.Ya. Kolbasov. A Facile Electrochemical Method for Graphene Nanoplatelets Preparation Using Multi-walled Carbon Nanotubes // FUEL CELLS, 2019, pp. 1–9. DOI: 10.1002/fuce.201800082
5. S.S. Fomanyuk, V.O. Smilyk, G.Ya. Kolbasov, I.A. Rusetskiy. Optical properties of NiOOH films in formaldehyde solutions. // Chemical Papers, 2019, pp. 1–9. DOI:10.1007/s11696-019-00900-z
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## Abstracts

1. Русецький І.А., Данилов М.О., Фоманюк С.С., Щербакова Л.Г., Патлун Д.В., Колбасов Г.Я., Солонін Ю.М. Синтез електродних матеріалів на основі оксидів, халькогенідів, ванадатів металів та графеноподібних сполук для фотоелектрохімічної системи з отриманням водню. Розробка конструкції фотоелектрохімічної комірки з акумулюванням водню та повітряним електродом. // Тези доповідей наукової звітної сесії “Розвиток наукових засад отримання, зберігання та використання водню в системах автономного енергозабезпечення”. – 11 грудня 2019, Київ. – С. 16.
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**Thank you for attention**