

Original Article

# Synthesis of Zinc Phthalocyanine Pigment and its Application to New Generation Solar Cells

Dilmurod Shukurov<sup>1</sup>, Khayit Turaev<sup>2</sup>, Bakhodir Kholnazarov<sup>3</sup>, Sherzod Kasimov<sup>4</sup>, Zulkhumor Jumaeva<sup>5</sup> and Kholmamat Tillayev<sup>6</sup>

<sup>1,2,3,4,5,6</sup>Faculty of Chemistry, Termez State University, Termez, Uzbekistan

<sup>1</sup>Corresponding Author : [dkhursanovich@mail.ru](mailto:dkhursanovich@mail.ru)

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**Abstract** - In this work, aspects of the synthesis of zinc phthalocyanine pigment and its use in new third-generation solar cells were studied based on its physical and chemical properties. Based on the results of IR-spectrum, SEM and elemental analysis of the synthesized zinc phthalocyanine pigment, its analysis data are presented. Also, the photosensitizing properties of zinc phthalocyanine pigment, the SEM image of titanium dioxide coated on one side on a special transparent glass, and the current and voltage values obtained by dye-sensitized solar cells based on zinc phthalocyanine pigment were presented.

**Keywords** - Zinc phthalocyanine, Pigment, Photosensitizer, Titanium dioxide, Dye-sensitive solar cells.

## 1. Introduction

Phthalocyanines containing metal and non-metal atoms as the central atom have been traditionally used on an industrial scale for many years. They have been used since ancient times as paints and pigments, especially color printing inks, as pigments in varnishes, and are one of the main materials used to paint plastics, metal structures, and synthetic fibers. In addition, the valuable photophysical and semiconducting properties of phthalocyanines allow for many promising projects in the future [1].

The most important advantages of organic semiconductors based on phthalocyanines are their good optical and electrical properties, the availability of manufacturing capabilities, the absence of negative environmental effects, and economic efficiency. Currently, the semiconducting property of metal phthalocyanines is used to create new-generation solar cells, obtain energy harvesting materials, and obtain materials used in the sensitivity determination devices of gas sensors based on phthalocyanines [2]. Complexes of phthalocyanines that actively absorb electromagnetic radiation in the visible and near-infrared ranges can be used to create complex semiconductor structures that combine the properties of organic and inorganic semiconductors [3].

Recently, it has been shown that interesting two-dimensional structures can be created from phthalocyanine molecules for the production of novel gas-sensing sensors, energy converters, and catalytic membranes [4]. Also, molecular structures based on phthalocyanines and their properties are used as a nanocomposite input or thin film to create thin film transistors, semiconductor sensors, photoconverters and other semiconductor devices. Thus, due to the unique combination of valuable properties in phthalocyanines, derivatives of metal phthalocyanines are

widely used in many areas of modern materials technology [5].

Metal phthalocyanines, as well as copper and zinc phthalocyanine dyes, due to their unique photochemical properties, are important for us as sensitizing dyes in LCDs, that is, liquid crystal-based screens, laser printers, and most importantly, in solar cells based on sunlight-sensitive dyes [6]. Phthalocyanines, like polymer molecules, serve as the basis for a new generation of organic semiconductors, metal polymers. Phthalocyanines containing metal and non-metal atoms are used in the production of photosensitizers in all branches of modern microelectronics and nanotechnology [7].

The synthesis and research of metal and non-metal phthalocyanines and metal phthalocyanines, which form a bond with phthalanhydride as ligands and retain heterocyclic compounds, is of great importance in the future development of many new types of semiconductor and photoanode materials. Phthalocyanines do not interact with strong acids and alkalis and also have significant optical absorption in the infrared and visible parts of the spectrum [8].

Currently, research scientists are involved in the synthesis and research of complex molecular complexes based on the properties of phthalocyanine, as well as how the electrical and optical properties of phthalocyanine complexes change as a result of increasing the number of macrocycles. By introducing functional groups that bind to ligands in the metal-containing phthalocyanine molecule and the optical properties that occur in complex organic semiconductors, we are conducting scientific research in such areas as the description of photodynamic and electrophysical processes [9].



## 2. Experimental Part

### 2.1. Material and Methods

Synthesis of zinc phthalocyanine pigment was carried out according to the common method of obtaining metal phthalocyanine dyes. The following chemicals and equipment are required for the preparation of materials: zinc chloride, phthalic anhydride, urea, concentrated sulfuric acid 96%, dimethylformamide, n-methyl pyrrolidone, distilled water, a thermometer and a thermostable glass.

### 2.2. Synthesis of Zinc Phthalocyanine Pigment

We needed zinc chloride, urea, phthalic anhydride, and catalysts to synthesize zinc phthalocyanine pigment. 2.72 g (0.02 mol') of zinc chloride, 11.84 g (0.08 mol') of phthalic anhydride, 9.6 g (0.16 mol') of urea and 1% of the total catalyst in a 400 ml thermostable beaker was mixed thoroughly by adding ammonium heptamolybdate. When the temperature in the preheated oven reached 250°C, we put the reaction mixture in the beaker in the oven for 2.5 hours. Then, the colorless reaction mixture reacts under the influence of temperature and turns into a brown powdery

mass. It was removed from the oven, cooled to room temperature and dissolved in 96% concentrated sulfuric acid solution while stirring for 30 minutes. Then, this reaction mixture dissolved in concentrated sulfuric acid was filled to the full volume of the beaker and mixed again by pouring boiling water. Then, the initial products, which did not fully enter the reaction, melt and pass into the solution and leave the reaction medium.

At the end of the process, after additional cleaning, melting and neutralization, blue-green crystals appear and begin to sink to the bottom of the glass. The obtained precipitate is washed repeatedly with water and precipitated and neutralized with an ammonia solution, then filtered in a Buyuchner funnel and washed several times with distilled water. The washed product was dried in a drying oven at a temperature of 60°C for 12 hours. The dried blue pigment is soluble in dimethylformamide, dimethyl sulfoxide and N-methyl pyrrolidone. The yield of the obtained product was 85%. Figure 1 below shows the formation reaction of zinc phthalocyanine pigment.

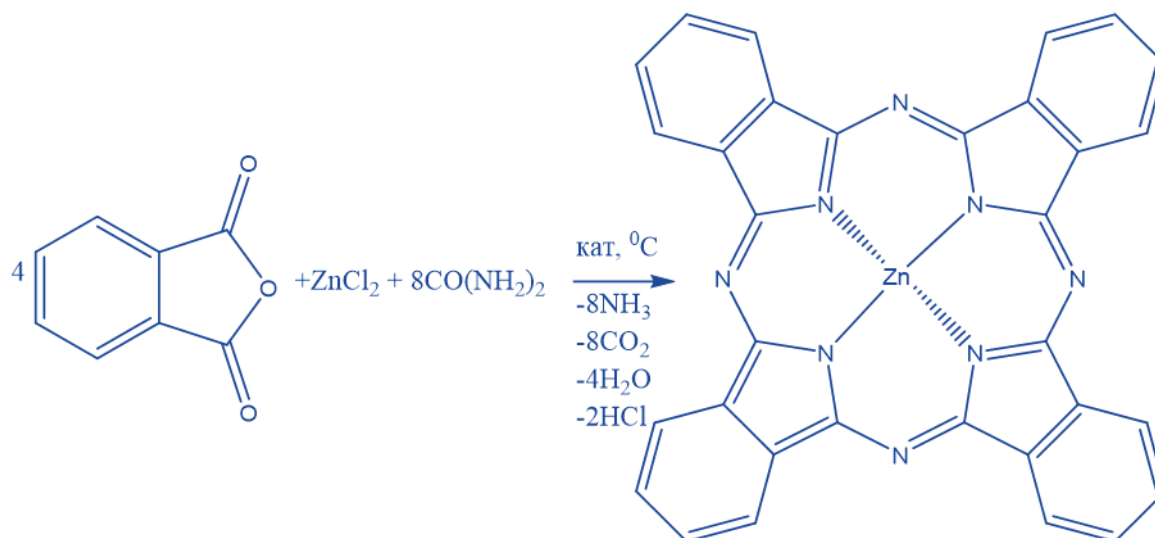


Fig. 1 Zinc phthalocyanine (ZnPc) pigment formation reaction

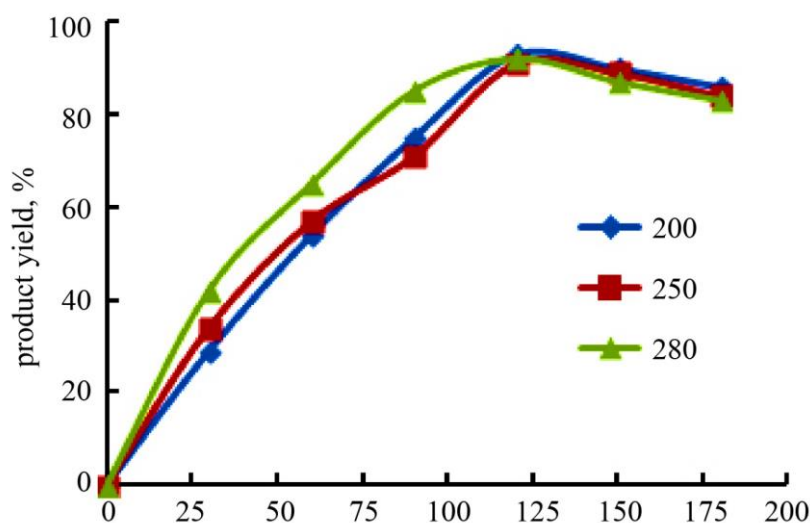


Fig. 2 Effect of temperature on product yield in zinc phthalocyanine (ZnPc) pigment synthesis process

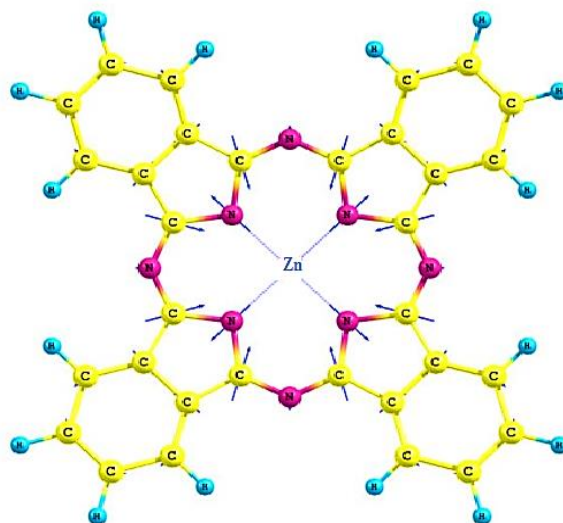


Fig. 3 Spherical structure of zinc phthalocyanine pigment

We identified the synthesized zinc phthalocyanine pigment by comparing it with the information in the literature and performing a physico-chemical analysis. Its excellent optical and photochemical properties distinguish the newly synthesized photoconductive dye substance. Also, its thermal stability, sensitivity to sunlight, and stability in solution expand its fields of application. Currently, we are carrying out scientific research on the application of this (ZnPc) zinc phthalocyanine pigment as a solar-sensitized dye for third-generation solar cells, which is one of the alternative energy sources.

### 2.3. Study of the Effect of Temperature on the Synthesis of Zinc Phthalocyanine (ZnPc) pigment

Temperature plays an important role in the pigment having high intensity and active properties. At the same time, if the high temperature exceeds the norm, the product yield will decrease to a certain extent, but the intensity will be high.

Pigment synthesis was carried out at 200, 250 and 280 °C. As shown in Figure 2, a temperature of 280 °C was selected for intensive, active pigment synthesis, where the maximum yield of the reaction in two hours was 92%, and after three hours, the yield decreased to 83%. The difference between the two yields showed that the 83% yielded pigment had higher voltage and current values, i.e. higher capacities resulting from the formation of dye-sensitized solar cells.

## 3. Results and Discussion

### 3.1. Study of the Physical and Chemical Properties of the Synthesized Zinc Phthalocyanine (ZnPc) Pigment

The amazing photophysical property of phthalocyanines containing metal and non-metal atoms as the central atom, i.e., field effect: by controlling the electrophysical parameters of a solid surface with the help of an electric field applied along the surface, they are widely used in the field of creating organic semiconductor devices.

Among metal phthalocyanines, zinc phthalocyanine and its derivatives are distinguished by having the necessary photophysical properties. The state of a long life of excited electrons, very good solubility in organic solvents, and high thermal stability are of great importance for this field. Zinc phthalocyanine and its derivatives, like copper and cobalt phthalocyanines, serve as the main components of hybrid systems as sensors and electrocatalytic materials in solar energy conversion devices. The conductivity property of organic semiconductors based on metal phthalocyanines is closely related to the mobility value of charge carriers.

Conductivity is one of the most frequently measured parameters when studying the properties of phthalocyanine-based thin films. Its initial value depends on many factors, such as polyamorphic modification, degree of crystallinity, the orientation of crystals, temperature, and electrophysical properties of the surrounding atmosphere. Phthalocyanine films greatly impact nature and the atmosphere in environment. Therefore, due to the high sensitivity of metal phthalocyanine films to the environment, they are widely used to produce chemical sensors for the atmosphere.

In the technology of creating organic photovoltaic materials, acting as a component of hybrid structures, their molecules are coated on the surface of various substrates and serve as a functional unit in the resulting device. The coating of phthalocyanines on the surface is usually covalent, non-covalent interaction using the capabilities of the aromatic system or due to external influences. In phthalocyanines, due to the effect of mutual  $\pi$ - $\pi$  bonding of electrons, retention and adsorption on the surface is strong. If positively charged functional groups are included, they can interact electrostatically with the electron surplus (negatively charged) structures of certain carbon materials.

Under certain conditions, phthalocyanines containing metal or non-metal atoms as central atoms and their

derivatives can interact with carbon materials graphene, graphene oxide and oxide substrates (TiO<sub>2</sub>, ZnO, SiO<sub>2</sub>, etc.).

The main purpose of phthalocyanines in such hybrid structures is to efficiently absorb light energy into the substrate and transfer the charge to the part that converts electricity. In this regard, phthalocyanine molecules used in photovoltaic materials act as photosensitizers to ensure the transfer of charges from the electron donor to the acceptor.

### 3.2. Infrared Spectroscopic Analysis of Zinc Phthalocyanine Pigment

Figure 4 shows the absorption spectra of the zinc phthalocyanine assembly in the near 400 cm<sup>-1</sup> and far 4000 cm<sup>-1</sup> infrared wavelength regions. The value of absorption

frequencies in organic semiconductors depends on the properties of the substrate material used. Absorption spectra of the zinc phthalocyanine complex in the near 400 cm<sup>-1</sup> and far 4000 cm<sup>-1</sup> infrared wavelength regions are shown. Absorption lines in the region of 3057.42 cm<sup>-1</sup> were found to belong to the S-H bond. The vibrations related to the C=C bond were observed to belong to the absorption lines at 1603.38 cm<sup>-1</sup>. It was determined that the absorption lines of the aromatic ring correspond to isoindole in the areas of 1486-1467 cm<sup>-1</sup>. It was observed that the valence vibrations with absorption peaks at 1357.72 cm<sup>-1</sup> belong to the pyrrole family. The absorption area of benzene rings at 947 cm<sup>-1</sup>, as well as absorption areas belonging to phthalocyanine rings, was observed in the region of 745.36 cm<sup>-1</sup> [10].

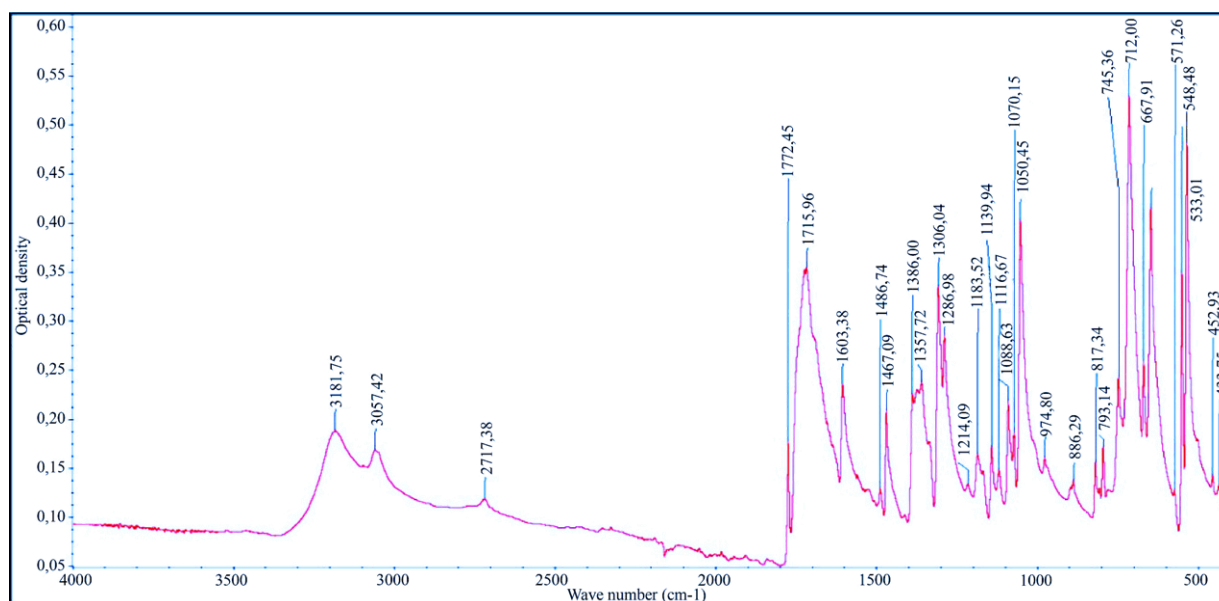


Fig. 4 IR spectrum of zinc phthalocyanine (ZnPc) pigment

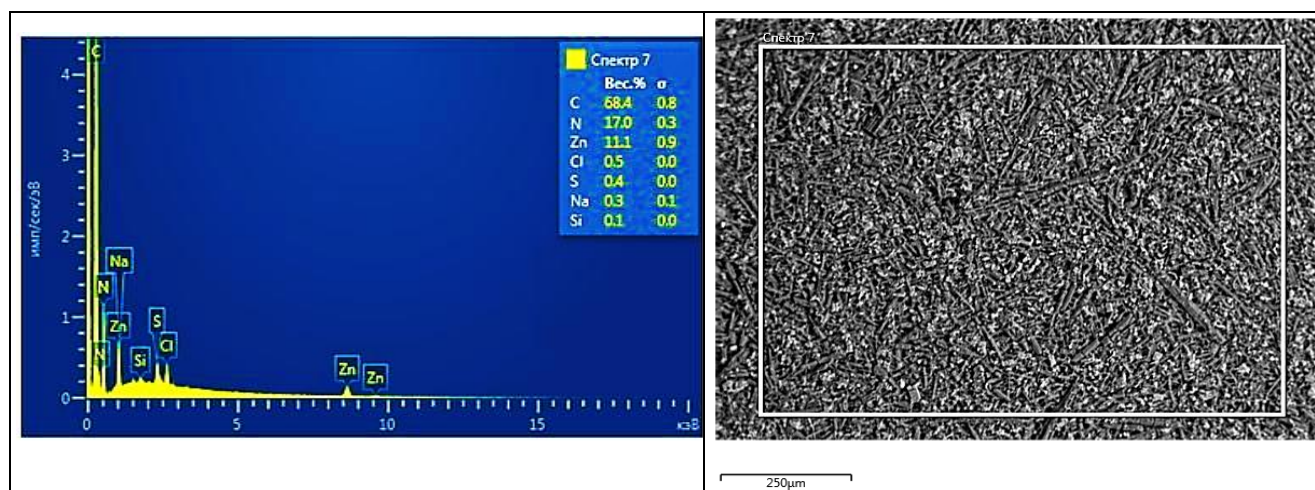


Fig. 5 Elemental and SEM analysis data of zinc phthalocyanine pigment

The elemental composition of the substance includes the raw materials used in any production and must be known to control the products. Considering this, element analysis was carried out on a separate surface, even in large clusters. Elemental analysis of large clusters showed that in addition

to the zinc phthalocyanine dye, chlorine, silicon, oxygen, and sulfur residues were left over from the substance used as a catalyst in the incompletely purified reaction and the substance used in neutralization.



**Table 1. Results of elemental analysis of zinc phthalocyanine (ZnPc) pigment**

Element	C	N	Zn	O	Cl	S	Na	Si
Quantity, %	68.38	17.05	11.11	2.3	0.52	0.36	0.25	0.07

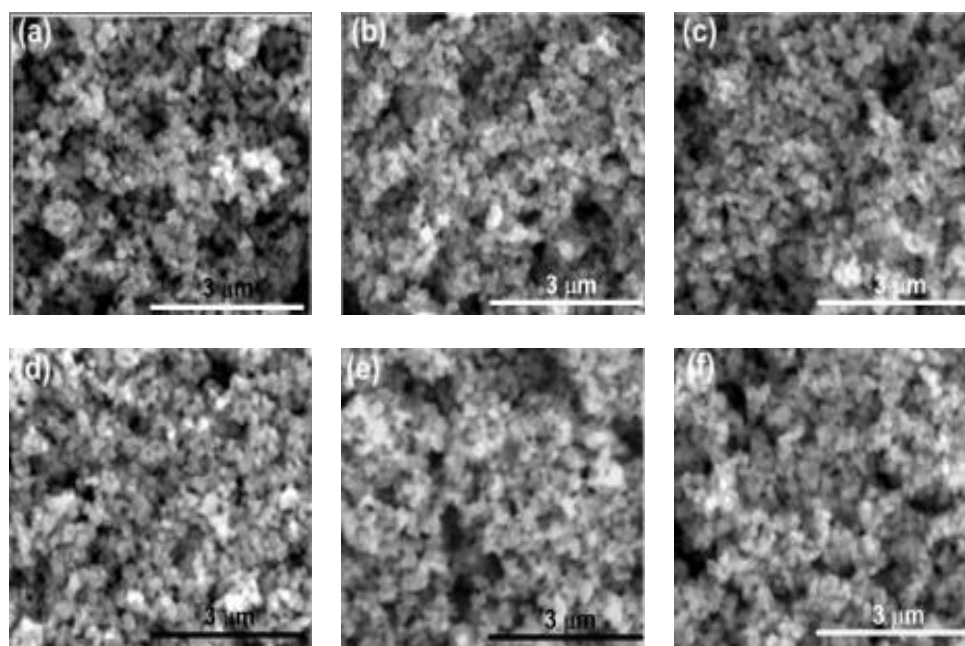
### 3.3. The composition and Preparation for the use of Single-Layer Special Transparent Glasses used in the Construction of Solar Cells based on Solar-Sensitive Paints

It is composed of indium and tin oxides. The chemical formula is  $\text{In}_2\text{O}_3$  90% 0.9 and  $\text{SnO}_2$  10% 0.1. Indium tin oxide (ITO), for short, is a semiconductor material that is transparent to visible light due to its large band gap (about 4 eV) but can reflect IR radiation. It is an n-type semiconductor material comparable to metals, in which tin ions serve as electron donors [30].

These materials provide a unique combination of high electrical conductivity with optical transparency in the visible wavelength range. The conduction band position of ITO is favorable for electron transport. Transparent conductive oxides have high optical and electrical conductivity. They are widely studied for use in devices used in contact formation as glass electrodes for electrical and optical applications, such as flat panel displays (FPDs), light-emitting diodes (LEDs), and photovoltaic solar cells. They are transparent in visible and near-infrared light and

have a high level of electronic conductivity and a high brightness coefficient. It is also used in solar cells based on solar-sensitive dyes for its excellent properties, such as complete adhesion to the substrate, optical absorption, hardness and chemical inertness [12].

It achieves high transparency in thin layers of 200 nm thickness deposited on a special transparent glass at a temperature of about 450°C. In dye-sensitized solar cells, as a conductive glass substrate, i.e., (FTO) fluorine-coated tin oxide, zinc-coated aluminum oxide (AZO), graphene for conducting polymers, and vanadate oxides as semiconducting transparent material are used. The dye solution is placed in a petri dish to sensitize the semiconductor material. The titanium dioxide-coated (ITO) electrode is immersed in the dye solution for 2 hours to absorb the dye into the active sites of  $\text{TiO}_2$ . Then, it was washed with distilled water and ethanol and then dried in the open air. The ITO transparent electrode reflects infrared rays like a metallic mirror, which allows it to be used for thermal protection and, in turn, for obtaining superconducting coatings from other materials [13].



**Fig. 6 SEM image of titanium dioxide coated on one side of special transparent glass**

To achieve high performance of DSSC, a large surface area of the  $\text{TiO}_2$  layer nanostructure is important because it allows the adsorption of a sufficiently large number of dye molecules necessary for efficient light conversion [15,33]. To reduce the reaction of photogenerated electrons with

electrolyte tri-iodide and guarantee good electrical conductivity, the following are required:

- large surface area of the  $\text{TiO}_2$  layer;
- good bonds between  $\text{TiO}_2$  grains;
- high adhesion to (ITO) -layer.

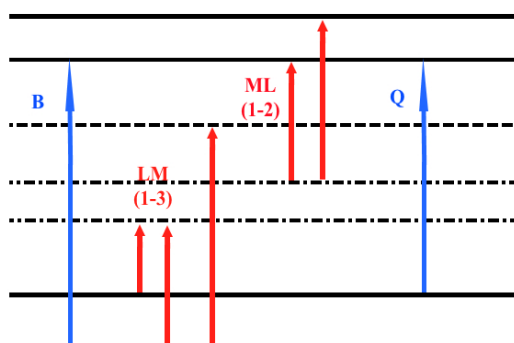
### 3.4. Optical Properties of Phthalocyanines

Absorption spectra of metal phthalocyanines have two very dense bands in the 300-400 nm and 650-700 nm regions. In addition, the shape and position of peaks in the absorption spectrum can change depending on the composition of peripheral and central terms or substituents and specific properties of the structures. When the semiconductor metal, phthalocyanine pigment molecules absorb light from the sun, electrons transfer from the highest energy-occupied molecular orbital (HOMO) level to the lowest energy-occupied molecular orbital (LUMO) due to the interaction between the orbitals of this pigment dye and the energy networks of the semiconductor should be obtained [16, 34].

HOMO and LUMO are the highest occupied molecular orbitals and the lowest occupied molecular orbitals, respectively, and are the zones of motion of excited electrons Scheme 1. In the scheme below, in the regions (B) and (Q), the direction of movement of electrons in the highest and lowest absorption regions, that is, the energy difference between HOMO and LUMO, or the HOMO-LUMO gap, is sometimes called frontier orbitals [18,19]. The electron transition energy difference between these two frontier orbitals can be used to predict the response of the optical properties of metal phthalocyanines to incident light, as well as the intensity and stability of pigments and their colors in solution [20,21].

Electron transition between HOMO and LUMO orbitals, that is, the direction of movement of excited electrons through the zones, is determined by the metal in the central atom and the substituted radical molecules on the edges of the phthalocyanine ring. An electronic transition between these states creates two excited states. The metal on the central atom and the molecular groups on the edges of the ligands affect the HOMO and LUMO transition energies. The above approaches are used in the physics and chemistry of phthalocyanines to describe their semiconducting and photosensitizing properties [22, 23].

Phthalocyanine molecules exhibit excellent optical absorption properties for visible and infrared light. In the literature, most metal phthalocyanines show very good absorption spectra in the optical absorption region between 400 nm and 700 nm [24].



Scheme 1. Movement of excited electrons through energy levels in metal phthalocyanines

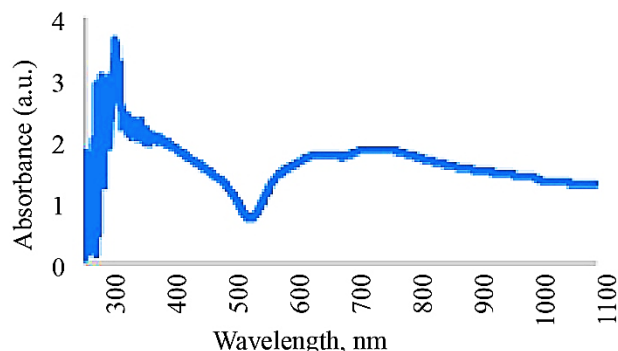


Fig. 7 Ratio of light absorption to the wavelength of zinc phthalocyanine pigment in UV-spectrophotometer

In addition, their absorption spectra may differ depending on the type of substituent radicals on the central atom and ligands. These absorption regions are important materials in terms of optical broadening [25].

Figure 7 shows the results of measuring the zinc phthalocyanine pigment in the UV-Vis spectrophotometer under the visible light spectrum from 300-1100 nm. Zinc phthalocyanine pigment was found to have high absorbance in the visible wavelength range of 500 nm to 600 nm. The wavelength with the highest absorbance of 530 nm was observed. As a result, copper phthalocyanine pigment was selected as a dye for dye-sensitized solar cells due to its ability to absorb photons from light. In the literature, several natural pigments have been used as dyes for dye-sensitized solar cells [26]. Taking this into account, we compared and analyzed the voltage and current values obtained based on pitahaya (cactus, dragon tree) fruit extract with our synthesized pigment. Medical pigments have a high absorption level but are not stable in aggressive environments and have a high tendency to corrode [11].

### 3.5. Photosensitizing Properties of Inorganic and Organic Compounds

After silicon-based solar cells convert solar energy into electricity at high efficiency, the most studied inorganic and organic solar cells are solar cells with solar-sensitive dyes. The process is supported by sunlight with titanium dioxide coated on a special transparent glass plate, and a sensitive paint sensitizer adsorbed on it. The photosensitizing property appears on the surface of titanium dioxide interacting with the relevant dye groups, such as carboxylate, phosphonate, as well as imide, imine or hydroxyl groups in the phthalocyanine ring in metallic or non-metallic organic dyes [28].

Sunlight is absorbed by dyes placed in the pores of the titanium dioxide microlayer coated on a transparent special glass plate. As a result of the polarization of sunlight, introducing electrons from dyes into the microlayer of titanium dioxide causes its photoconductivity. Solar cells based on solar-sensitive dyes are systems that convert visible light into electrical energy; whereby etching sensitive dyes into a coating of titanium dioxide, the dye absorbs photons from solar energy and converts the visible light into electrical energy. It is necessary to test the photosensitizing

properties of different dyes to solve the problems of efficient photoelectric conversion. Phthalocyanines and pentacenes are important in this regard [29, 36].

**3.6. Principle of Operation of Dye-Sensitized Solar Cells**

When sunlight passes through conductive glass, a dye (photosensitizer) sensitive to sunlight (visible) light absorbs the photon. The dye electron will be excited and injected into the conduction band of the TiO<sub>2</sub> and flow through the external wire to the load (e.g. lamp). The loss of an electron

from the dye will be compensated by an electron from the electrolyte as a result of the redox reaction of iodide (I<sup>-</sup>) oxidized to triiodide (I<sub>3</sub><sup>-</sup>) and the release of an electron. The electron from the load will flow back into the DSSC through the Pd/Au coated counter electrode (ITO), which catalytically promotes the reduction of electrolyte triiodide (I<sub>3</sub><sup>-</sup>) to iodide (I<sup>-</sup>) and is reused in the light to electrical energy conversion process in the DSSC. Previously, the principle of operation and properties of this solar cell were carefully studied by Gratzel and O'Regan with staff [31, 32].

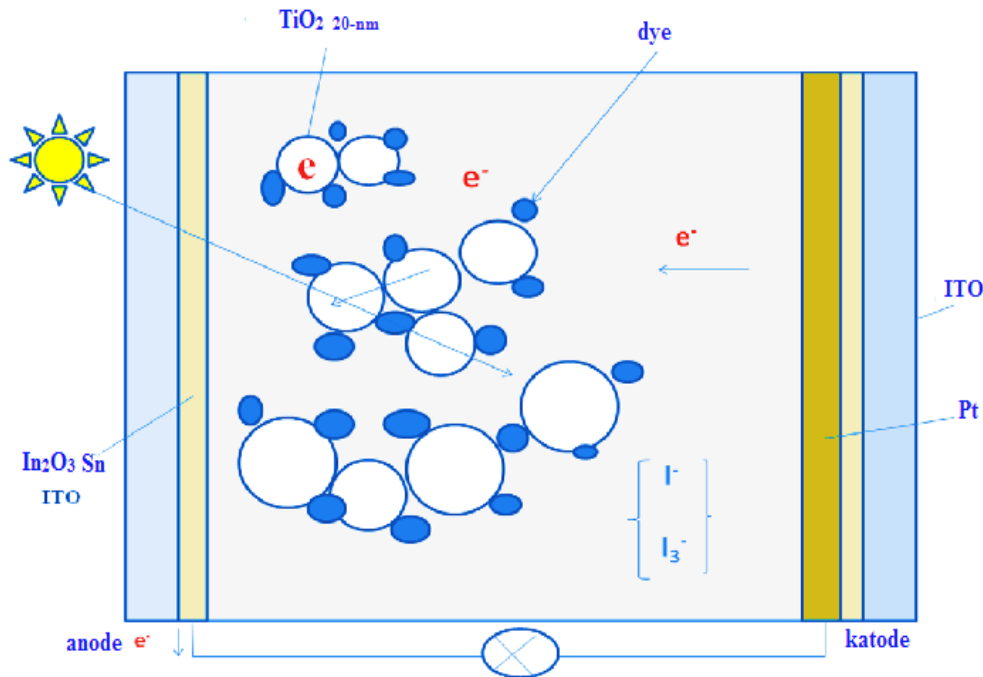


Fig. 8 General view of dye-sensitive solar cells (DSSC)

Table 2. Voltage and current values generated by pitahaya fruit extract with zinc phthalocyanine used as a dye in solar cells based on solar-sensitive dyes

Capacities obtained based on zinc phthalocyanine pigment			Strengths obtained based on pitahaya (cactus, dragon tree) fruit extract		
№ Time, day	Voc (mV)	Isc (mAcm <sup>-1</sup> )	№ Time, day	Voc (mV)	Isc (mAcm <sup>-1</sup> )
1	689,72	89.7	1	400	63
2	685,3	86.5	2	385.1	56.3
3	664	74,9	3	360.2	44.2
4	586	68.5	4	345.4	40.6
5	568	62.3	5	321.3	37.2
6	543	60.1	6	300.6	26.4
7	531,6	53.8	7	286.4	20
8	476	37.5	8	250.6	17.8
9	450	35.2	9	236	14.6
10	338	30.6	10	233,2	12.1

For comparison, solar cells based on sensitive dyes obtained from pitahaya fruit extract obtained from the literature have the highest voltage value of 400 (mV) and the lowest value of 233.2 (mV) and the highest current value of 63 (mA) as the lowest value. as compared to 12.1 (mA) results. The zinc phthalocyanine pigment we synthesized is stable in aggressive environments, and the generated voltage and current strength values are higher than natural pigments compared to natural pigments.

#### 4. Conclusion

The synthesized zinc phthalocyanine pigment formed was analyzed by infrared analysis, element analysis spectrum analysis in SEM and comparison with existing phthalocyanines. According to the results of the elemental analysis of the obtained pigment, it was found that it contains 11.11% zinc. Also, the obtained zinc phthalocyanine

pigment was dissolved in n-methyl pyrrolidone, and its optical absorption spectra, i.e. photodynamic properties, were studied in a UV-spectrophotometer. Due to the good ability of this pigment to absorb photons from the sunlight, the dye-sensitized solar cells yielded a maximum voltage of 400 (mV) and a minimum of 233.2 (mV) and a maximum current of 63 (mA). ) reported results of 12.1 (mA) as the lowest. Our ongoing intensive research aims to increase the useful duty ratio of zinc phthalocyanine pigment (ZnPc) for future solar cell, photoanode and energy harvesting applications.

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