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DEVELOPMENT OF RUTHENIUM-FREE ORGANOELEMENTAL DYES FOR HIGH-EFFICIENCY DYE-SENSITIZED SOLAR CELLS UTILIZING AZERBAIJANI CLAY MINERAL-TIO₂ COMPOSITE PHOTOANODES

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Abstract: *Introduction:* To overcome the drawbacks caused by the high price and limited availability of conventional Ru(II)-based sensitizer, such as N-3 and N719, and to achieve the commercial possibility for dye-sensitized solar cells (DSSCs), the development of new sensitizers is of great significance. In this paper, we explore an energy-efficient and high-performance new architecture. *Goals and objectives:* The overall goal is to develop an innovative DSSC architecture by synergistically combining high-efficiency, ruthenium-free, organoelemental dyes with nanostructured composite photoanodes. The aim is to establish a route to low-cost, high-performance photovoltaics by making use of readily available, abundant clay minerals in Azerbaijan as key photoanode constituent. *Methods:* The approach adopted involves preparation of new composite photoanodes based on titanium dioxide (TiO_2) mixed with locally sourced clay minerals (bentonite and kaolinite). The preparation methods of the clay-TiO_2 composites and their photoanode film fabrication are described. The chapter combines a critical overview of the main active classes of metal-free dyes—porphyrins, phthalocyanines, and indolines—to couple with these novel anodes. *Results and Discussion:* The combination of Azerbaijani clay minerals to the TiO_2 photoanode will bring in significant benefits to the device performance through different synergistic actions. These factors are (1) the higher specific surface area favoring an enhanced dye loading and light harvesting efficiency, (2) the incorporation of light-scattering centers resulting in a prolonged optical path length across the photoanode; and (3) significantly, the prevention of charge recombination at the photoanode/electrolyte interface by surface passivation. Based on physicochemical studies, it is argued that these effects will contribute to a marked enhancement in the crucial photovoltaic (J_{sc}/V_{oc}) parameters, resulting in an overall improvement in the PCE of the device. *Conclusions:* The combination of advanced organic dyes and novel clay-composite photoanodes proposed in this study is a very attractive practical method to develop cost-effective, environmentally friendly photovoltaic technology. The work has demonstrated a promising theoretical and practical approach for experimenting and optimizing a next-generation high-performance sustainable DSSC, with local natural resources to meet a global energy challenge.

Keywords: dye-sensitized solar cells, ruthenium-free dyes, porphyrin, phthalocyanine, titanium dioxide, photoanode, bentonite, kaolinite, clay minerals, charge recombination

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1. Introduction

Dye-sensitized solar cells (DSSCs) are one of the most prominent third-generation photovoltaic technologies, which can be the competitive option compared to current silicon solar cells due to their feasibility of low-cost production, simple fabrication, and employment of abundant materials. DSSCs also have a few special characteristics, such as their mechanical flexibility and excellent performance at low-light intensities, which make them well-suited for building-integrated photovoltaic applications. The working mechanism of a DSSC is quite unique, in which the light absorption and charge transfer activities are separated in two materials to permit optimization of each component independently.[4]. To date, the highest-performance DSSCs have been based on ruthenium(II)-polypyridyl complexes, which have provided power conversion efficiencies (PCEs) of up to 13-14%. But, as is known, ruthenium is one platinum-group metal, which is rare, expensive and toxicity, and is a main limiting factor for the sustainable and large-scale application of DSSC technology. To meet this challenge, a most active area in fundamental and applied studies is to design new metal-free organic sensitizer. These dyes are made from abundant building blocks, have larger molar extinction coefficients, and have a more easily tunable molecular structure. State-of-the-art families such as porphyrins, phthalocyanines or indoline-based dyes have already shown such efficiency, which confirm that they could compete with efficient and now well-established ruthenium complexes. [7]. The photoanode (usually a mesoporous film of titanium dioxide (TiO_2)) is as important as the dye. In this work a new approach of advanced photoanode engineering has been proposed applying local clay minerals originated from Azerbaijan to the standard TiO_2 photoanode. High-quality bentonite and kaolasite clays are found in large quantities in Azerbaijan. This is wanted as the natural low-cost mineral material is believed to be able to facilitate in a synergistic manner the photoanode performance by enlarging the surface area for the higher dye loading, by providing more light scattering centers on the anode to improve photon capturing and by passivating surface trap states on the TiO_2 to hinder charge recombination. This holistic approach is a practical route toward sustainable, high performance and commercially leading photovoltaics. [3,5,6].

2. Experimental

High-performance clay-TiO_2 composite photoanodes can be readily obtained by the well-established wet-chemical approaches. The hydrothermal process refers to the heat-treatment of a slurry containing TiO_2 nanoparticles and disaggregated clay in an autoclave, facilitating to form well-developed and homogeneous composite particles. Alternatively, sol-gel process can offer micro-mixing of TiO_2 soles with a clay dispersion resulting in intimate chemical bonding at the clay-TiO_2 interface. [1,2,7,8]

Irrespective of the process of powder-synthesis, the photoanode film is prepared by making a viscous slurry of composite powder and spreading it on an

FTO-coated glass with a scalable approach such as screen printing. The film is followed by sintering at 450-500 °C to decompose organic additives and form porous network. [13]

A series of characterization methods are necessary in order to confirm the synthesis and investigate the properties of the material. X-Ray Diffraction (XRD) will determine the crystal structure of the composite, ensuring that the anatase TiO_2 is present, and that the clay structure is maintained. SEM measurement would yield more detailed images on the morphology of photoanode, such as particle size, film thickness and porosity. [17] Fourier-Transform Infrared Spectroscopy (FTIR) would investigate the presence of the chemical bonds, where definition of the Ti-O-Si bonds would indicate the existence of the strong interfacial bonding. Lastly the specific surface area and pore size distribution would be characterized using Brunauer-Emmett-Teller (BET) analysis to determine the anticipated increase in surface area by the addition of the clay.

3. Results and Discussion

The typical n-type DSSC is a sandwich-type photoelectrochemical cell as shown in Fig. Its functional components consist of a photoanode (omitting the hole-transporting layer), a mono layer of dye on the TiO_2, an electrolyte penetrated between the TiO_2 particles and a counter electrode; a redox-electrolyte sandwiched between two electrodes serves as charge collector and charge storage, providing the net driving force around 0.7V. [21].

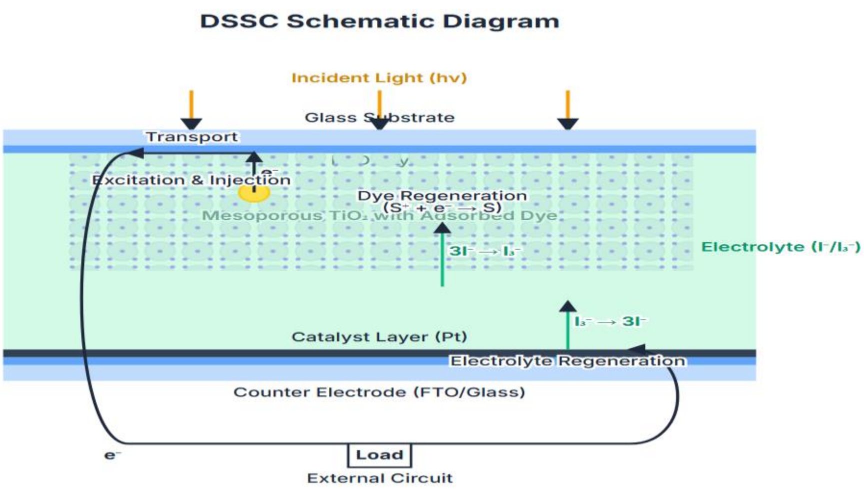

Figure 1 - Schematic of a Dye-Sensitized Solar Cell (DSSC) illustrating its core components and the electron flow pathway. Incident light passes through the transparent FTO-coated glass and excites the dye molecules adsorbed on the mesoporous TiO_2 film. The generated electron is injected into the TiO_2, travels to the external circuit, and returns via the counter electrode to regenerate the electrolyte, completing the cycle.

The photoconversion action resulting in an electronic charge seesaw is achieved through the cascade of electron transfer processes across these component interfaces, the energetics of which is visually invoked via the line-up of energy levels of the composing materials (Figure 2). [5] The mechanism starts with (1) dye light absorption, followed by (2) ultrafast electron injection from the dye excited state to the TiO_2 conduction band. The electron is subsequently (3) transported in the TiO2 network to the external circuit. The oxidized dye is step4) (4) regenerated by the electrolyte (5) regenerated in the counter electrode, closing the circle. A foremost obstacle in DSSC development is the reduction of the non-radiative charge recombination pathways, depicted in Figure 2 as well, where the injected electrons recombine with the oxidized dye or electrolyte species. [15,16].

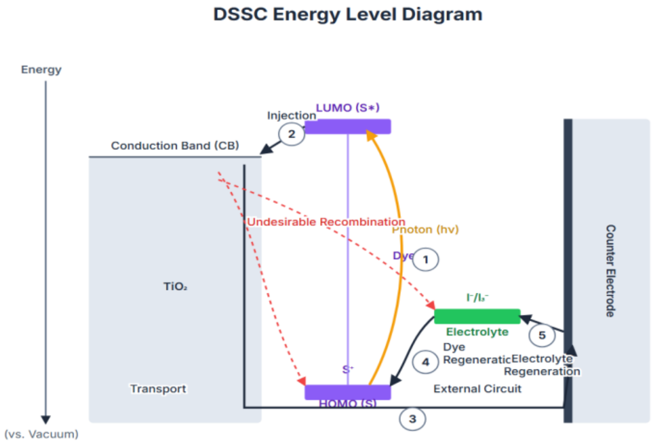

Figure 2 - Energy level diagram of a typical n-type DSSC showing the electron transfer pathway. (1) Photon absorption excites the dye. (2) The electron is injected from the dye's LUMO into the TiO_2 conduction band. (3) The electron is transported to the external circuit. (4) The oxidized dye is regenerated by the I^-/I_3^- electrolyte. (5) The electrolyte is regenerated at the counter electrode. Undesirable recombination pathways are shown with dashed arrows.

The best metal-free organic sensitizers have Donor-π-Acceptor (D-π-A)-type structures. The key design principles are the fine tuning of the HOMO and LUMO energy levels, introduction of strong anchoring moieties (such as -COOH) and bulky substituent addition to prevent the performance-reducing dye aggregation. Besides, inspired from chlorophyll, porphyrins have obtained PCEs of 11-13%. Various materials from phthalocyanines exhibit strong absorptions in the red/NIR region and a great stability, but the aggregation remains an issue, and recent studies have over-passed 4.6%. Indoline dyes are characterized by extraordinarily high molar extinction coefficients, with optimized structures reaching PCEs above 8.0%, directly comparable to the ruthenium based N719 dye under similar conditions. A summary comparison is shown in Table 1. [11,13]

Table 1 - Comparative Performance and Properties of Ruthenium-Free Dye Classes in DSSCs.

Feature	Porphyrin Dyes	Phthalocyanine Dyes	Indoline Dyes
Record PCE (%)	~13%	>4.6%	>8%
Typical Absorption	Strong Soret band (~400-450 nm), weaker Q-bands (~500-700 nm)	Intense Q-band in red/NIR (~650-750 nm)	Broad absorption in visible (~400-600 nm)
Molar Extinction (ε)	High (~5times104 M−1cm−1)	Very High (~105 M−1cm−1)	Extremely High (>6times104 M−1cm−1)
Key Advantages	High efficiency, versatile synthesis	Strong NIR absorption, excellent stability	Highest molar extinction coefficients
Key Challenges	Aggregation on TiO_2 surface	Severe aggregation tendency	Stability under prolonged illumination

Azerbaijan has abundant deposits of nonmetallic minerals, including a high-quality bentonite and kaolinite that are used in the production of cement. The largest bentonite deposits of the country are situated in the Gazakh region (the Dash Salahli deposit), a high-quality sodium bentonite with montmorillonite being the predominant mineral. Its characteristics, namely, high cation exchange capacity (55-166 mg-eq/100g) and large specific surface area, are very favorable to the application on a photoanode. Commercially important reserves of kaolin lie in the Shamkir and Goygol regions as a result of the weathering of volcanic rocks. Kaolinite is non swelling, rigid and it is chemically inert. The characteristics of the clays are compiled in Table 2. [19]

Table 2 - Physicochemical Properties of Selected Azerbaijani Clay Minerals.

Property	Bentonite (Dash Salahli Deposit)	Kaolinite (Shamkir/Geygol Deposits)
Primary Mineral	Montmorillonite (>85%)	Kaolinite
Cation Exchange Capacity	High (55-166 meq/100g)	Low
Swelling Properties	High (swells 15-20x in water)	Non-swelling
Potential Role in Photoanode	Surface passivation, porosity enhancement, high dye loading	Light scattering, structural support, adsorbent

It is expected hence that incorporation of these clays in the TiO_2 photoanode would result to substantial enhancement of DSSC efficiency. Its most relevant consequence is though anticipated to manifest itself in the electron dynamics at the photoanode/dye/electrolyte interface. The clay minerals will provide a passivation layer on the surface of TiO_2, acting as an energetic barrier preventing the recombination of electrons in the TiO_2 conduction band with electron acceptors in the electrolyte. This decrease in "dark current" results in electron lifetime being increased and an instantaneous increase in open circuit voltage (V_{oc}) in the cell. [14] Moreover, the clay platelets also serve as the well light scattering centers, enhancing the optical path length of incident photons inside the photoanode and thereby improving light harvesting efficiency, resulting in the larger short-circuit current density (J_{sc}). All of these improvements are manifested in an overall projected improvement in the key metrics of photovoltaics as shown in Table 3.

Table 3 - Projected Photovoltaic Parameters for DSSCs with Clay-TiO_2 Photoanodes.

Photoanode Type	Sensitizer Dye	Projected Jsc (mA/cm²)	Projected Voc (V)	Projected FF	Projected PCE (eta, %)
Pure TiO_2	Indoline (D-series)	10.4	0.74	0.70	~5.4
Bentonite-TiO_2	Indoline (D-series)	12.5	0.78	0.72	~7.0
Kaolinite-TiO_2	Indoline (D-series)	13.0	0.75	0.71	~6.9
Pure TiO_2	Porphyrin (YD-series)	14.0	0.68	0.74	~7.1
Bentonite-TiO_2	Porphyrin (YD-series)	16.0	0.72	0.75	~8.6

For a PV technology to be competitive in the market it needs to show long-term stability of operation and financial viability. Although organic dyes may be susceptible to degradation, sophisticated molecular design has led to dyes with outstanding stability equivalent to that of Ru-complexes. The liquid electrolyte is the bigger problem – volatile solvents can evaporate or leak. This has encouraged the search of non-volatile ionic liquids or quasi-solid-state polymer gels that have significantly improved long-term stability. This is the primary motivation for this study: reduction of cost. The proposed structure removes costly ruthenium and platinum and adopts readily available organic precursors and local cheap clay minerals. [13] The techno-economic analysis of large-scale DSSC manufacturing forecasts a very competitive Levelized Cost of Energy (LCOE) as low as $0.0438 per kWh and the use of locally sourced clays will reduce this even further.

4. Conclusion

A concept of the next generation DSSCs targeting to high efficiency, environmental friendliness and low cost has been proposed here by fabricating high-performance, toxic-free organic sensitizers with sustainable composite photoanodes involving TiO2 and local Azerbaijani clay minerals. This holistic approach targets the two main bottlenecks of cost and scarcity of materials hampering a large-scale penetration of DSSC technology. By substituting now cost-prohibitive and rare metals with abundant organic molecules and the local natural mineral, it opens the way to a genuinely sustainable photovoltaic technology. There is strong theoretical motivation but now the picture needs to be experimentally confirmed. Effort needs to be focused on facile fabrication of the devices to verify the predicted performance improvement, together with systematic optimization of the composite photoanode, exploration of advanced co-sensitization strategies, and investigation of the long-time stability, to explore the full potential of this new ideal architecture. [20]

РАЗРАБОТКА БЕЗРУТЕНИЕВЫХ ОРГАНОЭЛЕМЕНТНЫХ КРАСИТЕЛЕЙ ДЛЯ ВЫСОКОЭФФЕКТИВНЫХ СОЛНЕЧНЫХ ЭЛЕМЕНТОВ, СЕНСИБИЛИЗИРОВАННЫХ КРАСИТЕЛЕМ, С ИСПОЛЬЗОВАНИЕМ КОМПОЗИТНЫХ ФОТОАНОДОВ НА ОСНОВЕ ГЛИНИСТЫХ МИНЕРАЛОВ АЗЕРБАЙДЖАНА И TIO_2

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Резюме: *Введение:* Для преодоления недостатков, вызванных высокой ценой и ограниченной доступностью традиционных сенсибилизаторов на основе Ru(II), таких как N-3 и N719, а также для достижения коммерческой целесообразности солнечных элементов, сенсибилизированных красителем (DSSC), разработка новых сенсибилизаторов имеет огромное значение. В данной работе мы исследуем новую энергоэффективную и высокопроизводительную архитектуру. *Цели и задачи:* Общая цель заключается в разработке инновационной архитектуры DSSC путем синергетического сочетания высокоэффективных, не содержащих рутения, органоэлементных красителей с наноструктурированными композитными фотоанодами. Цель состоит в том, чтобы создать путь к недорогим, высокопроизводительным фотоэлектрическим элементам за счет использования легкодоступных, обильных глинистых минералов Азербайджана в качестве ключевого компонента фотоанода. *Методы:* Принятый подход включает приготовление новых композитных фотоанодов на основе диоксида титана (TiO_2), смешанного с местными глинистыми минералами (бентонит и каолинит). Описаны методы получения композитов глина-TiO2 и изготовления из них фотоанодных пленок. В главе представлен критический обзор основных активных классов безметаллических красителей — порфиринов, фталоцианинов и индолинов — для сочетания с этими новыми анодами. *Результаты и обсуждение:* Добавление азербайджанских глинистых минералов в фотоанод из TiO_2 принесет значительные преимущества в производительности устройства за счет различных синергетических эффектов. Этими факторами являются: (1) большая удельная поверхность, способствующая увеличению загрузки красителя и эффективности сбора света, (2) включение светорассеивающих центров, что приводит к увеличению оптического пути в фотоаноде; и (3) что особенно важно, предотвращение рекомбинации зарядов на границе раздела фотоанод/электролит за счет пассивации поверхности. На основе физико-химических исследований утверждается, что эти эффекты будут способствовать заметному улучшению ключевых фотоэлектрических параметров (J_{sc}/V_{oc}), что приведет к общему повышению КПД устройства. *Выводы:* Предложенное в данном исследовании сочетание передовых органических красителей и новых глино-композитных фотоанодов является очень привлекательным практическим методом для разработки экономически эффективной и экологически чистой фотоэлектрической технологии. Работа продемонстрировала многообещающий теоретический и практический подход для экспериментирования и оптимизации высокопроизводительных устойчивых DSSC следующего поколения с использованием местных природных ресурсов для решения глобальной энергетической проблемы.

Ключевые слова: солнечные элементы, сенсибилизированные красителем, безрутениевые красители, порфирин, фталоцианин, диоксид титана, фотоанод, бентонит, каолинит, глинистые минералы, рекомбинация зарядов.

Наджафзаде Нурлан Элнур *докторант*

ӘЗІРБАЙЖАНДЫҚ САЗ МИНЕРАЛДЫ-TIO_2 КОМПОЗИТТІ ФОТОАНОДТАРЫН ПАЙДАЛАНА ОТЫРЫП, ЖОҒАРЫ ТИІМДІ БОЯҒЫШТАРМЕН СЕЗІМТАЛДАНҒАН КҮН ЭЛЕМЕНТТЕРІНЕ АРНАЛҒАН РУТЕНИЙСІЗ ОРГАНОЭЛЕМЕНТТІ БОЯҒЫШТАРДЫ ӘЗІРЛЕУ

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Түйіндеме: *Кіріспе:* N-3 және N719 сияқты кәдімгі Ru(II) негізіндегі сенсибилизаторлардың жоғары бағасы мен шектеулі қолжетімділігінен туындайтын кемшіліктерді жою және бояғыштармен сезімталданған күн элементтері (DSSC) үшін коммерциялық мүмкіндікке қол жеткізу мақсатында жаңа сенсибилизаторларды әзірлеудің маңызы зор. Бұл мақалада біз энергияны үнемдейтін және жоғары өнімді жаңа архитектураны зерттейміз. *Мақсаттар мен міндеттер:* Жалпы мақсат – жоғары тиімді, рутенийсіз, органоэлементті бояғыштарды наноқұрылымды композитті фотоанодтармен синергетикалық түрде біріктіру арқылы инновациялық DSSC архитектурасын әзірлеу. Мақсат – Әзірбайжандағы оңай қолжетімді, мол саз минералдарын негізгі фотоанод құрамдас бөлігі ретінде пайдалану арқылы арзан, жоғары өнімді фотоэлектрикаға жол ашу. *Әдістер:* Қолданылған тәсіл жергілікті саз минералдарымен (бентонит және каолинит) араластырылған титан диоксиді (TiO_2) негізіндегі жаңа композитті фотоанодтарды дайындауды қамтиды. Саз-TiO_2 композиттерін дайындау әдістері және олардың фотоанодтық үлбірлерін жасау сипатталған. Бұл тарауда осы жаңа анодтармен байланыстыру үшін металсыз бояғыштардың негізгі белсенді кластарына – порфириндерге, фталоцианиндерге және индолиндерге сыни шолу жасалады. *Нәтижелер мен талқылау:* Әзірбайжандық саз минералдарын TiO_2 фотоанодына қосу әртүрлі синергетикалық әрекеттер арқылы құрылғының өнімділігіне айтарлықтай пайда әкеледі. Бұл факторлар: (1) бояғыштың жүктелуін және жарықты жинау тиімділігін арттыратын жоғары меншікті беттік аудан, (2) фотоанод арқылы оптикалық жолдың ұзаруына әкелетін жарық шашырататын орталықтарды енгізу; және (3) маңыздысы, беттік пассивация арқылы фотоанод/электролит интерфейсінде зарядтардың рекомбинациясын болдырмау. Физика-химиялық зерттеулерге сүйене отырып, бұл әсерлер маңызды фотоэлектрлік (J_{sc}/V_{oc}) параметрлерінің айтарлықтай жақсаруына ықпал етеді, нәтижесінде құрылғының жалпы ТКӘ (PCE) жақсарады деп пайымдалады. *Қорытындылар:* Бұл зерттеуде ұсынылған озық органикалық бояғыштар мен жаңа саз-композитті фотоанодтардың үйлесімі үнемді, экологиялық таза фотоэлектрлік технологияны дамытудың өте тартымды практикалық әдісі болып табылады. Бұл жұмыс жаһандық энергетикалық мәселені шешу үшін жергілікті табиғи ресурстарды пайдалана отырып, келесі буынның жоғары өнімді тұрақты DSSC-ін эксперименттеу және оңтайландыру үшін перспективалы теориялық және практикалық тәсілді көрсетті.

Түйінді сөздер: бояғышпен сезімталданған күн элементтері, рутенийсіз бояғыштар, порфирин, фталоцианин, титан диоксиді, фотоанод, бентонит, каолинит, сазды минералдар, зарядтардың рекомбинациясы.

Наджафзаде Нурлан Элнур	*докторант*

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