

**IZMIR KATIP CELEBI UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES**

**INCREASING THE EFFICIENCY OF DYE-SENSITIZED SOLAR CELLS
USING IONIC LIQUID BASED ELECTROLYTES**



M.Sc. THESIS

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Department of Nanoscience and Nanotechnology

Thesis Advisor: Assoc. Prof. Dr. Mehmet Ertuğrul SOLMAZ

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İZMİR KATİP ÇELEBİ ÜNİVERSİTESİ
FEN BİLİMLERİ ENSTİTÜSÜ

BOYA DUYARLILAŞTIRILMIŞ GÜNEŞ PİLLERİNİN VERİMLERİNİ
İYONİK SIVI ESASLI ELEKTROLİTLER KULLANARAK ARTIRMAK

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OCAK 2018

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To my family,



FOREWORD

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ABBREVIATIONS AND SYMBOLS

Ag	: Silver
Ar	: Argon
Al	: Aluminum
Cd	: Cadmium Sulfide
Cdl	: Capacitance of double layers
CIGS	: Copper indium gallium selenide
CV	: Cyclic voltammetry
CuPc	: Copper Phthalocyanine
D	: Diffusion coefficients
DMSO	: Dimethyl Sulfoxide
DSSC	: Dye sensitized solar cell
EIS	: Electrical impedance spectroscopy
FF	: Filling Factor
FT-IR	: Fourier Transform Infrared Spektrofotometer
H-NMR	: Hydrogen-nuclear magnetic resonance spectroscopy
HOMO	: Highest Occupied Molecular Orbital
IPCE	: Incident photon to current conversion efficiency
I	: Current
I_{sc}	: Short Circuit Current
I-V	: Current-voltage
J	: Current Density
J_{sc}	: Short Circuit Current
Li	: Lithium
LUMO	: Lowest Unoccupied Molecular Orbital
mM	: Milimolar
MPP	: Maximum power point
mW	: Milliwatt
P_{sp}	: Solar cell surface
R	: Resistance
R_{ct}	: Charge transfer resistance
R_{rec}	: Recombination resistance
Si	: Silicon
te	: Life times of electrons
TBAPF6	: Tetrabutylammonium Hexafluorophosphate
TiO₂	: Titanium Dioxide
UV-Vis	: Ultraviolet–visible
V	: Volt
V_{oc}	: Open Circuit Voltage
W	: Watt
Z	: Impedance

ZnO : Zinc oxide
 η : Efficiency



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ABSTRACT

In the last century, the energy demand of the world has been continuously increasing, and new and more environment-friendly energy sources search continues. As a result, Solar Cells have emerged as a renewable energy type. With the fact that when the sun is used efficiently it is a constant source of energy and a never-ending source of energy all over the world, triggering scientific studies on this subject and intensifying studies on solar batteries. With the influence of the economic factors in the materials, this issue has come up in three phases day by day. The fact that it is cheap, easy to procure, and that the organic dye-based structure is evolving with developing organic chemistry has made these devices attractive. Two factors that are important in the efficiency of these devices are the dye and the electrolyte. In these significantly developing systems, we have focused on recent studies on the use of ionic liquid based electrolytes. In this work, which we did, we concentrated on the ionic liquid electrolyte and tried to improve the efficiency. I-V characterizations of the produced DSSC devices were viewed and the performance related efficiency was investigated. As a result, ionic liquid based electrolyte has been shown to increase the efficiency of DSSC devices.



BOYA DUYARLILAŐTIRILMIŐ GÜNEŐ PİLLERİNİN VERİMLERİNİ İYONİK SIVI ESASLI ELEKTROLİTLER KULLANARAK ARTIRMAK

ÖZET

Son yüzyılda dünyanın enerji ihtiyacı sürekli artmaktadır ve de daha yeni ve daha çevreci enerji kaynağı arayışları sürmektedir. Bunun bir sonucu olarak, yenilenebilir bir enerji çeşidi olarak Güneş Pilleri öne çıkmıştır. Güneş'in verimli kullanıldığına tüm dünyadaki ihtiyaca hayli hayli yetecek ve hiç bitmeyen sürekli bir enerji kaynağı olması, bu konudaki bilimsel çalışmaları tetiklemiştir ve güneş pilleri çalışmaları yoğunlaşmıştır. Malzemelerdeki ekonomik etmenlerin etkisiyle bu konuda üç evrede günümüze kadar gelinmiştir. Ucuz olması, temininin kolay olması ve de organik boya esaslı yapısının gelişen organik kimya ile gelişime açık olması; bu organik boya esaslı güneş pillerini cazip hale getirmiştir. Bu cihazların verimlerinde önemli olan iki faktör kullanılan boya ve de elektrolittir. Önemli ölçüde gelişmekte olan bu sistemlerde, son dönemde iyonik sıvı esaslı elektrolitlerin kullanıldığı çalışmalar üzerine yoğunlaşmıştır. Biz de yaptığımız bu çalışmada iyonik sıvı elektrolit üzerine yoğunlaştık ve verimi geliştirmeye çalıştık. Üretilen DSSC aygıtlarının I-V karakterizasyonları incelenmiş ve de performanslarına ilişkin verimler araştırılmıştır. Sonuç olarak da iyonik sıvı esaslı elektrolitin DSSC aygıtlarının verimlerini artırdığı görülmüştür.



1. INTRODUCTION

In old times, energy was only used for heating and heating purposes, but today it has become almost the basic necessity of our daily life by the development of the electricity and the industry. It has become essential throughout life in eating, drinking, social life, communication, transportation and this has led to enormous energy needs. While this was met with simpler basic ways at first, it has become really difficult to meet the needs of the world with its increasing population and the electronicization of life. The need for energy has even become a cause of wars in the world. The pollution and the depletion of fossil fuel resources have led to a lack of cleaner renewable energy sources. Fortunately, the amount of energy coming from the Sun is 10000 times more than our current consumption. So; 0.1% of the world covered with solar batteries and 10% efficiency to operate the need for energy can meet the situation [1,2]. Solar power is the best choice for renewable energy and has a great potential for research. Solar cells can convert the energy coming from sun to electrical energy. The materials used in solar cell and their design affect the performance. Light can be defined as a flood of photons, and their energy is quantumized due to their colour or frequency. The matter that absorbs light, is transferring energy by photons and electrons increase their energy levels. Here, we transfer this energy to an electrical work by an external circuit [3].

In the 19th century, Edmund Becquerel succeeded in producing the current by inserting the electrode into the electrolyte and covering it with silver [4,5]. Further work was carried out by Siemens in 1883 [6]. Despite the fact that the cells at that time did not work very efficiently, this topic started to attract attention and became the beginning [7].

Based on photoelectric effect; Einstein's light is considered as quantities and formed by the description of the photoelectric phenomenon [8]. Until the 1930s, thin film with semiconductor-metal interface was concentrated on solar cells. Audubert and

Stora 1932 found the solar cell using cadmium sulphide (CdS) [9]. The present invention then opened the front of the II-VI systems and opened the silicon electronics. Later, many new studies were carried out on it and the charge distribution in the p-n joint was increased [10]. By using chapin selenium, it has begun to increase this yield even further [11]. Later on Berman's work on silicon, the interest in solar cells has increased considerably, as the price of a single crystal module has fallen and oil prices have increased at that time. In the 1990s, new systems called "third generation solar cells" were passed. The bulk layer in these new generations forms the basis of this, while the excited charges in the other solar cells were carried in p-n heterojunction [12].

1.1 Photovoltaic Effect

In 1839 Becquerel [4] discovered the photovoltaic effect in the experiment which he used the system that he found the first photovoltaic cell, photoelectrochemical solar cell. In Photo-electrochemical solar cell, a semi-conductor-electrolyte system is used as the photoactive layer. The first reports on such systems were published in the late 1960s. Unfortunately, semiconductors with the proper band gap are not stable and it was observed that it was undergoing corrosion. For this reason, such solar cells work is limited to broadband spaced semiconductors. This battery showed the expected stability, but yields it was low [13]. By the way, the high absorption properties of organic molecules were increasing their efficiency in using the sunlight. Organic polymers and molecules absorb light produce charges and transport these charges to the electrodes. Their ease of use and production and their high flexibility make them advantageous.

Firstly, in 1986, Tang used copper phthalocyanine (CuPc) compound in the organic solar cell with a yield of 0.9% [14]. After that, the new organic materials used have reached this yield of 6.5% [15]. However, in 1991, O'Regan and Grätzel achieved a yield of 7% with their photoelectrochemical battery system they formed nanocrystalline TiO₂ film sensitized with ruthenium bipyridyl complex dyes [16]. Ultimately, the use of liquid electrolyte achieved 11% efficiency [17]. However, the use of liquid electrolyte in the structure and its volatility are the disadvantages of these solar batteries. Although many studies have been made yet, the efficiencies

have not reached the desired level yet. Inorganic solar cells yield 25%. These are gallium arsenide-based inorganic solar cells and silicon-based single-junction solar cells [18].

Solar cells are semiconductor materials that absorb sunlight and convert them to electrical energy (Figure 1.1). It is the photovoltaic principle.

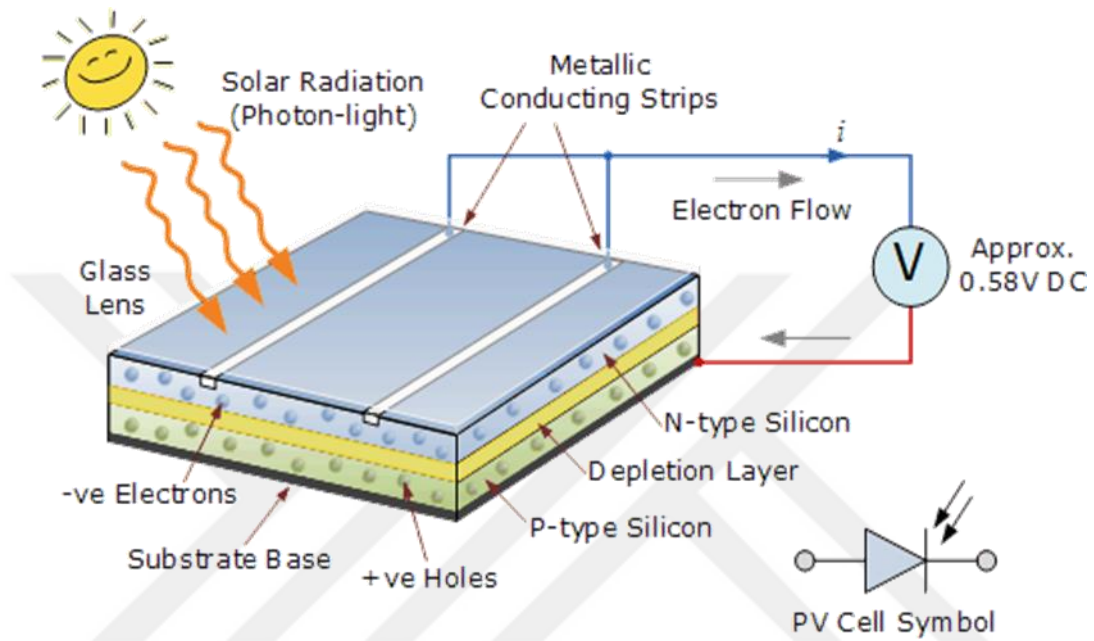


Figure 1.1 : Photovoltaic Solar Cell [19].

The source of electrical energy that the cell gives is the solar energy that comes to its surface. Photovoltaic transformation in the joint region makes them work as a photovoltaic cell. This happens in two stages. First, electron-hole pairs are formed by reducing the light to the joint region, and secondly, they are separated from each other by the help of the electric field in the region. Semiconductors consist of the Valence band and the Conductivity band. When a photon with energy equal to or greater than this forbidden energy range is absorbed by the semiconductor, it supplies its energy to an electron in the Valence band, allowing the electrons to reach the conduction band. Thus, an electron-hole pair is formed. If this phenomenon occurs at the interface of the pn junction photovoltaic cell, the electron-hole pairs are separated from each other by the electric field there. In this way, the photovoltaic cell works like a pump that pushes the electrons to the n (negative) region and the holes to the p (positive) region. The electron-hole pairs separated from each other, form a useful

power output at the ends of the photovoltaic cell. This process continues again with the impingement of a photon on the cell surface. In the inner parts of the semiconductor, electron-hole pairs are formed by incoming photons [20].

1.2 Dye-Sensitized Solar Cells

Dye-sensitized solar cells (DSSC) are a new type of solar cells, which have shown big improvements in this area. It is low cost because cheap materials are used, but some materials like ruthenium are slightly more expensive (Figure 1.2). DSSCs are separated from photovoltaic devices that use other Si semiconductors because of their efficiency and cost. DSSCs use a thin mesoporous metal oxide instead of a single crystal semiconductor, which is usually TiO_2 . TiO_2 is a good light absorber [21]. The use of liquid electrolyte can also cause problems in different weather conditions. In the early periods, ZnO semiconductors and rose bengal dye were used, but the efficiency was very low. The work of the Gratzel team in 1991 made a lot of progress and reached more than 7%. In these studies, TiO_2 was used as a semiconductor and a ruthenium based dye was used as light absorber [16]. It has been found that the use of a mesoporous semiconductor on the solar cell sensitized by the dye increases the surface area by about 1000 times. Semiconductor to be used is related to the dye used. TiO_2 still gives the highest efficiency.

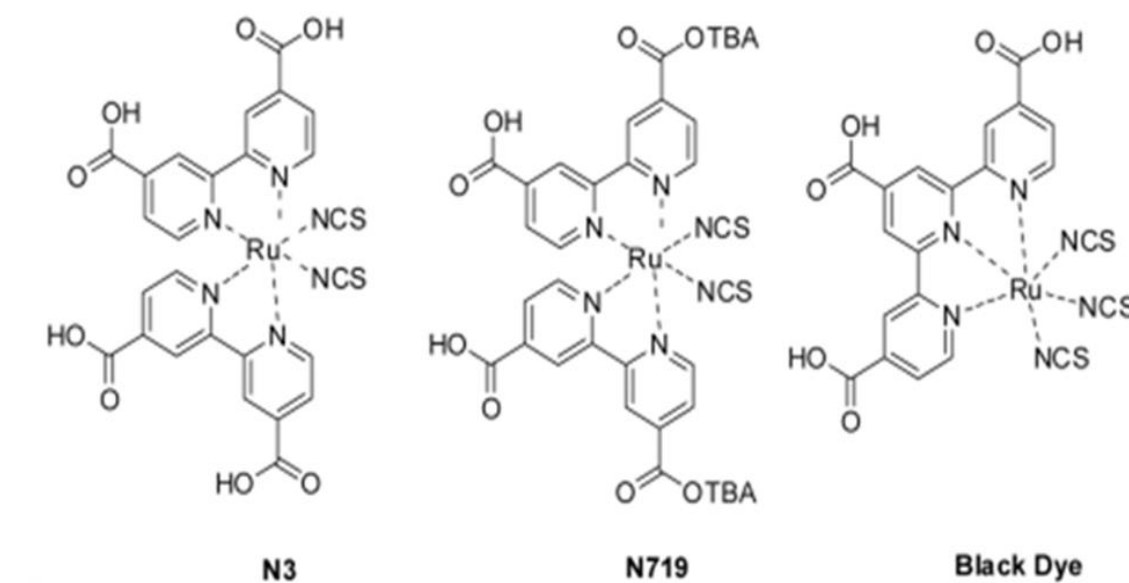
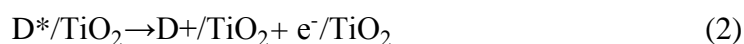


Figure 1.2 : Structures of the ruthenium-based dyes.

1.3 Structure of DSSC

Organic solar cell is a device that absorbs sunlight from the organic molecular layer and converts it directly to electrical energy. Organic solar cell works with organic polymers and small organic molecules to absorb electricity and produce electricity with photovoltaic effect. Organic dye based solar cell, in other words Grätzel cell, has a structure composed of three separate layers which are semiconductor, dye and electrolyte that transforms convert photon energy to electricity without any chemical transformation [22].

For DSSC to work, light has to be absorbed by dye molecules. The dye molecule (D) are excited (D*). Then the electrons stimulated by the dye are sent to the semiconductor TiO₂. They are stimulated in this way and send the electrons to the conduction band. These electrons are sent across this network at this point, reaching the electrode and entering the circuit. Finally, they are made neutral by the electrolyte [23].





With the energy generated by this movement of the electrons, the energy required for the operation of the electric devices is provided. In fact, the task of the dye in this circuit is the same as that of chlorophyll responsible for photosynthesis in green plants, so we can call it artificial chlorophyll (Figure 1.3).

Despite the effectivity and stability of these DSSCs, they are limited in some cases:

- Ruthenium dyes are expensive and water sensitive.
- Redox couple is performance limited.
- The electrolytes used are volatile.

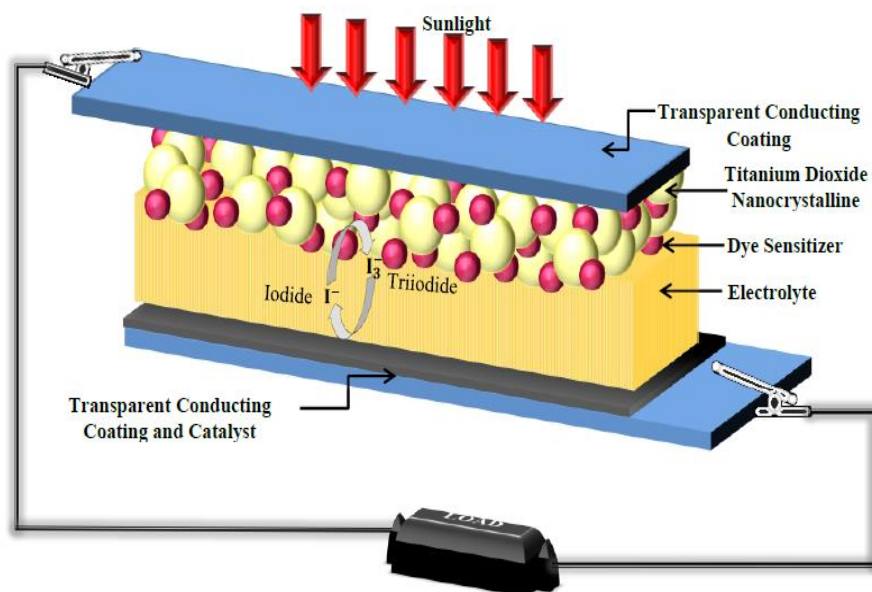


Figure 1.3: Principle operation of dye-sensitized solar cells [24].

1.4 Working Electrode

Different metal oxides are used in DSSC as working electrode. Of these, TiO_2 is most commonly used. 1991 was the beginning for that [16]. Titanium Dioxide

(TiO₂), which is a white synthetic pigment and applied to the paste-like cell. These TiO₂ type semiconductors not only act as dye sensitizers but also act as electron acceptors and electrical conductors. Other common semiconductor chemicals used in addition to TiO₂ are; gallium arsenic, silicon, copper oxide, indium and zinc oxide. Titanium dioxide (TiO₂) is a broadband, stable, non-toxic, high refractive index semiconductor. These structures are rutile, anatase and brokit. The first semiconductor material used in DSSC cells is ZnO. The conductivity band of ZnO and the E_g value is very similar to the anatase form of TiO₂. Although ZnO is a semiconductor with higher electron mobility than TiO₂, it exhibits a very weak character compared to TiO₂ in terms of chemical stability [7].

1.5 Dye

The most important feature of DSSCs is the organic dye they use. A single layer of the used dyes is adsorbed onto the surface of the semiconductor material by chemical bonding. Then the dye is excited by absorbing the incoming light. Once stimulated, it is oxidized by injecting electrons into the semiconductor and on the other hand electrons are removed from the redox couple in the electrolyte. Organic dyes have many advantages; such as higher damping, easier modification, and less harm to the environment when compared to transition metals. However, organic dyes have problems such as absorption in the narrow band, tendency to aggregate, and stability. One of the important recent studies to increase the yield of DSSC cells is the use of co-adsorbers. The most important feature of this molecule is that it does not absorb light [7]. At the beginning of these properties, low HOMO energy level and narrow band interval are required. Having a narrow band gap allows them to absorb in the near-infrared region [25].

Ruthenium polypyridyl dyes are not ideal dyes even though they are the most efficient dyes to date. Among these factors limiting these dyes are the difficulty of synthesis, the cost of the starting materials, the low coefficient of molar absorption and absorption in a very narrow range of the solar spectrum. In an organic dye-based solar cell, the dye adsorbed onto TiO₂ nanocrystalline particles must have some basic properties in order to provide high light-to-electric energy conversion.

Absorption: The dye must absorb in the visible region (400-700 nm).

Energy values: To reduce energy losses and keep the photovoltage at the highest possible level, the excited state energy of the dye should be just above the conduction band of TiO_2 and the energy difference must be sufficient to allow electron transfer.

Kinetics: Electron transfer from the excited state of the dye to the conductive band of TiO_2 must be very fast to avoid damping reactions such as fluorescence, phosphorescence or dark process

Stability: The dye, which has been adsorbed onto the TiO_2 surface, must be stable for long years at the working conditions (semiconductor-electrolyte interface).

Interfacial properties: Strong adsorption on the semiconductor surface.

Application Properties: The dye must have high solubility and contain a binding group that can be attached to the semiconductor surface [23].

1.6 Electrolyte

1.6.1 Electrolytes in dye-sensitized solar cells

Electrolytes are composed of two major species, organic solvents and ionic liquids, and this electrolyte are used in photovoltaic performances of DSSCs [26]. The electrolyte provides charge transfer between the electrodes and allows the dye to be regenerated. The electrolyte provides long stability, which affects conversion efficiency. Factors affecting DSSC yield are photo-current density (J_{sc}), photovoltage (V_{oc}) and fill factor (FF) [27]. J_{sc} is influenced by the redox couple charge transport. FF can be affected by the charge resistance between the electrolyte and the electrodes. And, the redox potential of the electrolyte affects V_{oc} . Therefore, the subject that is generally sensitive in this kind of scientific research has been electrolytes [28].

The electrolyte provides ionic conductivity between the electrodes in the DSSCs.

In DSSCs electrons are transported through the TiO_2 crystal film and the holes pass through the electrolyte and hole conductors. The role of the electrolyte and hole conductors is to regenerate the dye. This process, which takes place on TiO_2 , consists of several series of reactions. Electrolyte regeneration is terminated by the conversion of I_3^- ions to I^- [29].

1.6.2 Liquid Electrolytes

O'Regan and Gratzel in 1991 developed the first DSSC using liquid electrolyte without using an organic solvent and additive. They used iodide / triiodide redox couple and obtained a yield between 7.1 and 7.9% [16]. Liquid electrolytes are important with easy preparation, high conductivity, low viscosity and high efficiency. Liquid electrolytes are still the most used electrolytes for DSSCs and have reached up to 13% yield in conventional DSSCs [30].

Liquid electrolytes in DSSCs must be chemically and physically stable, must be low viscous to keep charge transport resistance low, and should be a good solvent for redox couples. The cell does not show much separation for the electrode. Usually; solvent, ionic conductor and additive [31].

1.6.3 Ionic Liquids

Ionic liquid is a liquid electrolyte consisting entirely of ions. Ionic liquids have anions and cations. The melting point is used to distinguish molten salts of high melting point and low melting point below 100 °C. Others are free-flowing liquids at room temperature, which are called room-temperature ionic liquids [28]. The most basic reason for the intensive use of ionic liquids in DSSCs is very low vapor pressures which allow low leakage and low evaporation. Also; good chemical and thermal stability, adjustable viscosity, relative incombustibility, high ionic conductivity.

There are two types of applications of ionic liquids in DSSC electrolytes. One is acting as solvent in liquid electrolytes and the other is acting as organic salt in quasi-solid-state electrolytes [32].

1.7 Aim of the Thesis

The place of DSSC cells in technology has an indisputable prefix. Significant developments were recorded by synthesizing and using different dyes from 1991 to the present day with the guidance of Gratzel. DSSC cells can not compete with commercially available silicon-based solar cells now. However, the efforts to synthesize new dyes are progressing rapidly, and as higher efficiencies in DSSC cells are reached, the usage rates are also rapidly increasing. In order to reach the desired efficiencies in the DSSC cells, the existing problems need solutions [13].

One of the aims of this thesis is to synthesize new ionic liquids with an aromatic structure containing imidazole groups which can be used as electrolytes in DSSC production and efficiently oxidized and reduced. And, we will calculate the performances of these DSSC

cells. In this context, three(3) ionic liquids with different molecular structures were synthesized. Since the molecular structure of these compounds are suitable to behave as electron sources, they can efficiently transfer electrons after excitation of the dye with the sunlight. And DSSC cells were fabricated. UV-Vis, cyclic voltammetry and current-voltage (I-V) measurements of the molecules were performed.



2. FABRICATION OF DSSC CELLS

0.5 mM dye solution was prepared with commercially purchased ruthenium based N719 dye and 100 ml Acetonitrile tertbutanol solvent (1: 1 by volume). Calculations made are as follows:

$$\text{Molarity(M)} = \frac{n(\text{mole})}{v(\text{L})}$$

$$0,5 \times 10^{-3} \quad n/100\text{mL}$$

$$n = 5 \times 10^{-5}$$

$$n = m/MA$$

$$MA = 1188.6$$

$$m = 59.93 \text{ g}$$

59.93 g of the powdered dye was added to the acetonitrile tertbutanol solvent. It was left on the ultrasonic cleaning device for 1 hour for complete dissolution. When the dye completely dissolved in the solvent, the dye solution became ready. It was kept in a dark environment.

TiO₂ electrodes, commercially purchased from Solaronix, were heated to a temperature of 500 °C in the oven to remove organics and contaminants. Electrolytes are placed in the oven which is at room temperature in a way that the TiO₂ surface does not contact the surface of the furnace. First, the temperature is increased to 100 °C. And the temperature is increased by 100 °C at each 5 minutes. Sudden temperature increases can crack the coatings. Lastly, it is left to cool in its own state at 500 °C waiting for 15 minutes.

When reaching 110 °C, the electrodes are removed from the oven (Figure 2.1(a)). They were immersed in dye when it was at 70-80 °C so that they do not adhere to the

TiO₂ surfaces (Figure 2.1(b)). The cups are closed and are kept overnight in the dark environment. An average of 12 hours is a matter of waiting. Electrodes that have been dyed throughout the night will become chemically diffused. Electrodes removed from the dye are immersed in acetone nitrite tertbutanol solvent, the solvent of the dye, and stored for at least 15 minutes. This is to ensure that the residues that have accumulated on the intended TiO₂ layer and are not chemically reacted are dissolved and removed from the electrode (Figure 2.2). Electrodes removed from the acetonitrile/-tertbutanol solvent are then placed in a container containing only acetonitrile and allowed to wait for 15 minutes. Electrodes that are removed are kept waiting for drying in room conditions. After drying well, the cell becomes ready for construction.



(a)



(b)

Figure 2.1: (a)The oven used, (b) the placement of TiO₂ in the oven.



Figure 2.2: Immersion in the solvent of the counter electrode.

TiO₂ coated electrodes are also called working electrodes. Once the working electrodes are ready, the platinum coated counter electrodes are heated to 500 °C in the oven to remove organics and remove them from the dirt. This heating is done step by step like in working electrode. Electrodes at room temperature are placed so that the platinum surface does not contact the surface of the oven. First, the temperature is increased to 100 °C. After waiting 5 minutes it is increased by 100 degrees every 5 minutes. Lastly, it is left to cool in its own state, waiting for 15 minutes at 500 °C. When reached 50 °C, the electrodes are removed from the oven. The platinum coated electrodes removed from the oven are ready to be joined with a TiO₂ coated electrode for DSSC construction.

For DSSC construction, the part to be worked on is thoroughly cleaned and laid out with a clean paper cover. The working electrode is turned over with the TiO₂ coated surface on top, and the Surlyn is placed on it. The counter electrode is positioned so that the perforated portion is on top of the TiO₂ coated electrode. The heater is then heated to 95 °C as shown in Figure 2.3. The assembled electrodes are placed so that the counter electrode is under the heater at 95 °C without moving and shifting. The Surlyn in the middle is melted and pressed with a wooden piece until it sticks well. Surlyn needs to be removed immediately when melted. Overheating can cause it to fully recover and is not a desirable event. If this happens, the coated layers will contact one another and cause a direct short circuit. Once this stage is complete, the

silver paste is ready for disposal so that it can be properly contacted with the ultrasonic soldering device. The surface of our cell coated with TiO_2 is soldered to the platinum coated surfaces using a silver paste to be contacted with the ultrasonic soldering device (Figure 2.4).

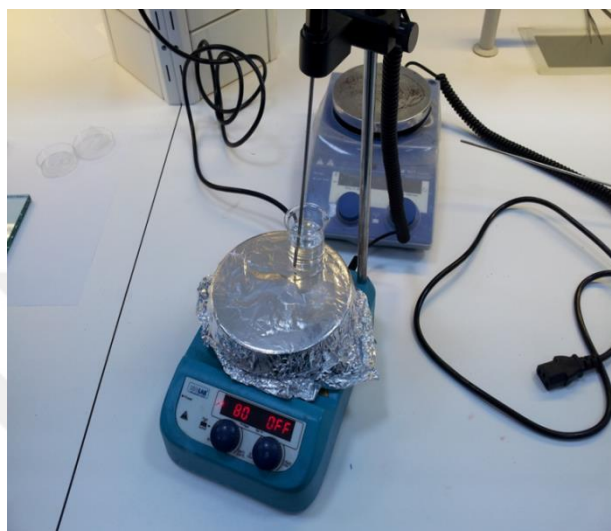


Figure 2.3: Heating for two electrodes.



Figure 2.4: Ultrasonic soldering construction stage.

The vacuum pump for electrolyte addition requires a thick glass to leave on the cell, and intermediate elements that allow the air inside the cell to be vacuumed. The arrangement is shown in Figure 2.5.

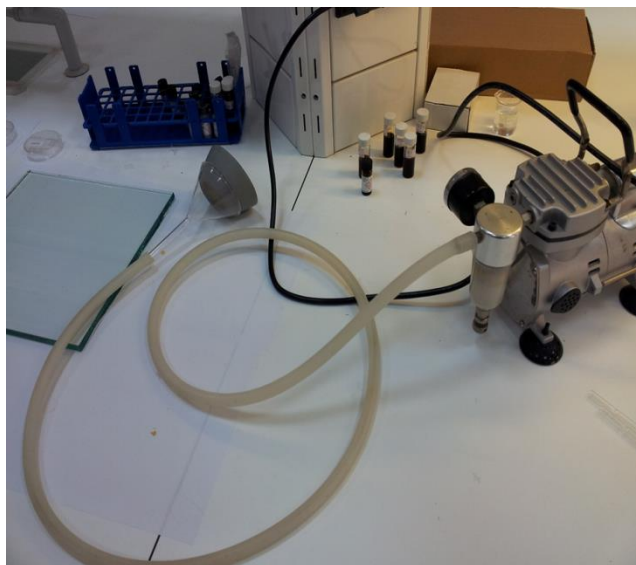


Figure 2.5: Vacuum Pump.

Iodine / triiodide commercially available from Solaronix is used as a chemical electrolyte. The glass pipette is covered with the electrolytic solution on the perforated part of the cell, then the vacuum is applied to remove the air between the electrodes. When the vacuum pump is turned off, the electrolyte enters the space between the air-drawn electrodes thanks to the outdoor air pressure. If the electrolyte does not penetrate fully, this process is repeated. Different pipettes are used every time to prevent contamination when electrolyte is taken. After each use, the cover of the electrolyte must be closed thoroughly. After the addition of the electrolyte is complete, the final stage of cell construction is ready.

In the last stage of our cell construction phase, surlyn is applied on the hole and a glass large enough is placed on it to cover the hole. This glass is pressed on with a heated iron tip and the surlyn is melted. The molten iron provides perforated glass with adhesion and closure of the thin glass we put on it. Now the DSSC has become ready for use (Figure 2.6).

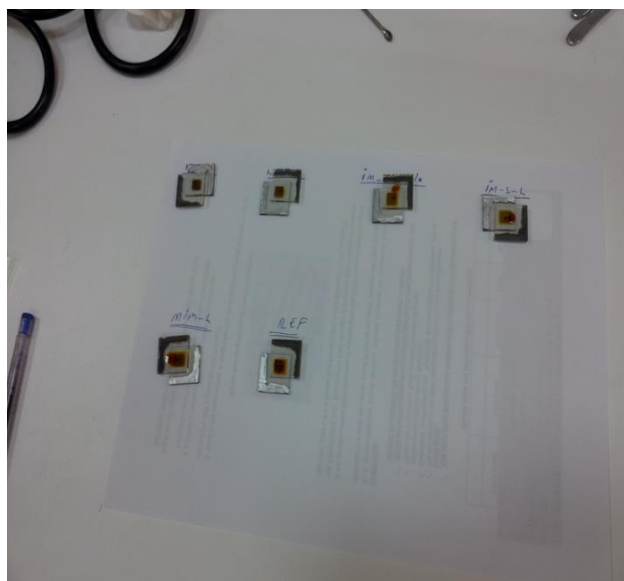


Figure 2.6: Prepared DSSCs.

3. RESULTS AND DISCUSSION

3.1 Synthesis and Structural Characterization

The synthetic route of the ionic liquids, standard liquid, MY1 and MY2 are depicted in Scheme 2. The asymmetric ionic liquids (MY1 and MY2) were prepared by the synthesis of the N-alkyl substituted imidazole ring of the imidazole followed by the reaction of the imidazole ring with the alkyl halide [33]. After completion of the synthetic works, the chemical structures of the ionic liquids were elucidated by FT-IR and $^1\text{H-NMR}$ spectroscopy.

3.2 Synthesis of Ionic Liquids

3.2.1 1-butyl-3-methyl-2,3-dihydro-1H-imidazolium iodide (Ref)

1-Methylimidazole (3.0 g; 36 mmol) and 1-iodobutane (7.4 g; 40 mmol) stirred at $110\text{ }^\circ\text{C}$ for 24 hours (Figure 3.1). The product was washed with diethylether (10mL) and recrystallized from dichloromethane/diethylether. The solvent was evaporated under vacuum and brown oil was obtained. Yield: % 72.

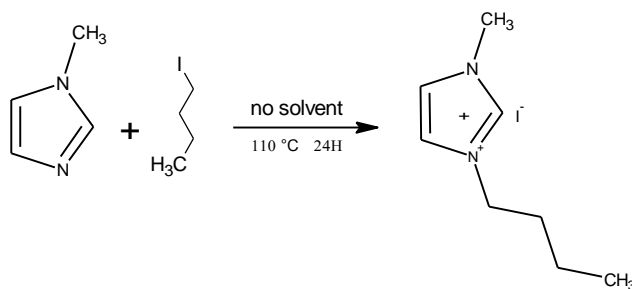


Figure 3.1: Stirring 1-Methylimidazole and 1-iodobutane.

And $^1\text{H-NMR}$ and FT-IR spectrums are given below in Figure 3.2 and Figure 3.3 respectively.

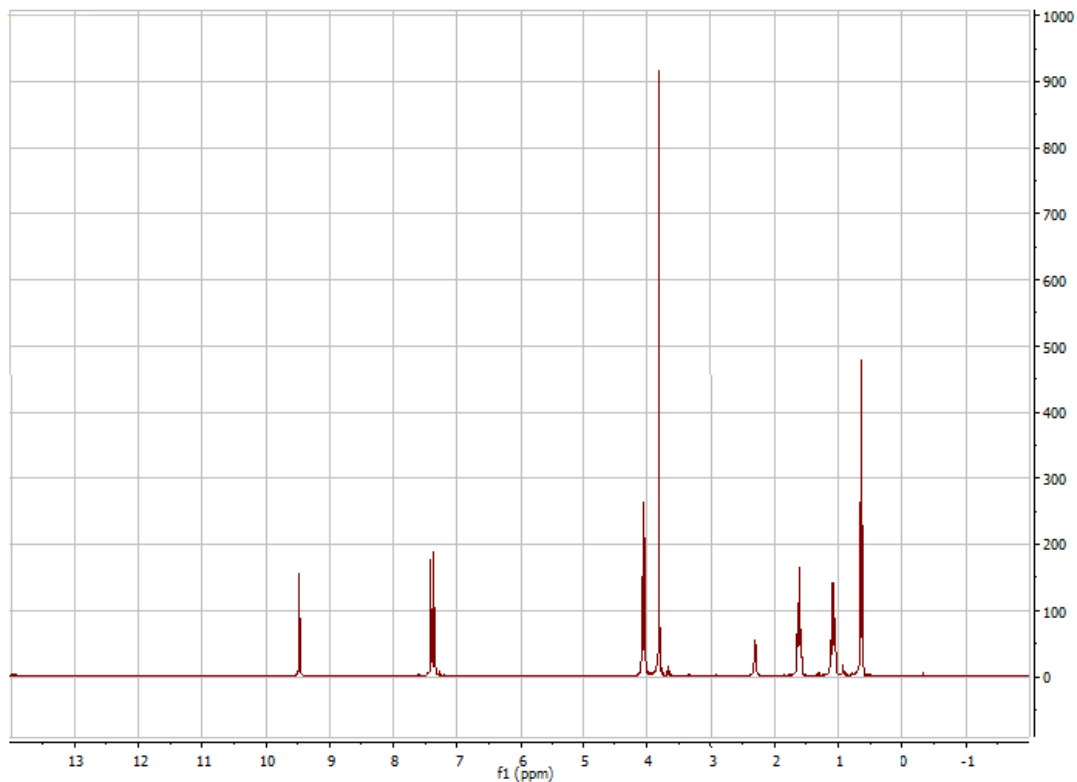


Figure 3.2: $^1\text{H-NMR}$ spectrum of Ref.

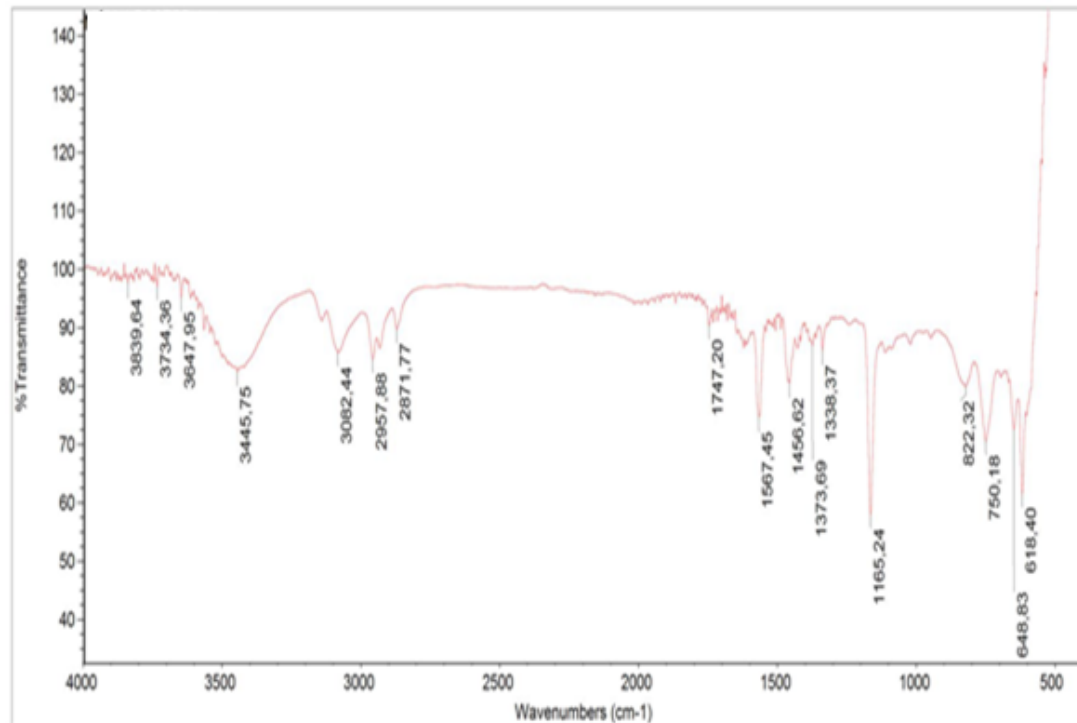


Figure 3.3: FT-IR spectrum of Ref.

1-butyl-3-methyl-2,3-dihydro-1H-imidazolium iodide (MY2)

Imidazole (5.00 g; 73.4 mmol) and KOH (6.16 g; 110 mmol) were dissolved in DMSO (10mL) and stirred at room temperature for 5 hours as shown in Figure 3.4. 2-Chloroethyl methyl ether (7.63g; 80.7mmol) was added. Mixture was stirred at 50 °C for 48 hours. After then, the reaction mixture was diluted with water (500mL) and extracted with CHCl₃ (5x25mL). Organic phases were combined and removed. The product washed with hexane and diethylether. After, solvents were evaporated under vacuum and orange oil formed.

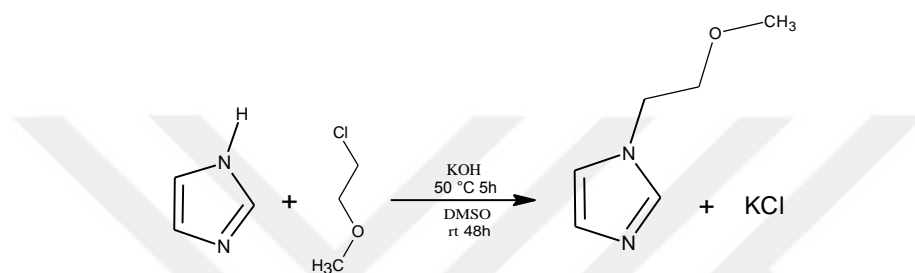


Figure 3.4: Dissolution of Imidazole and KOH in DMSO.

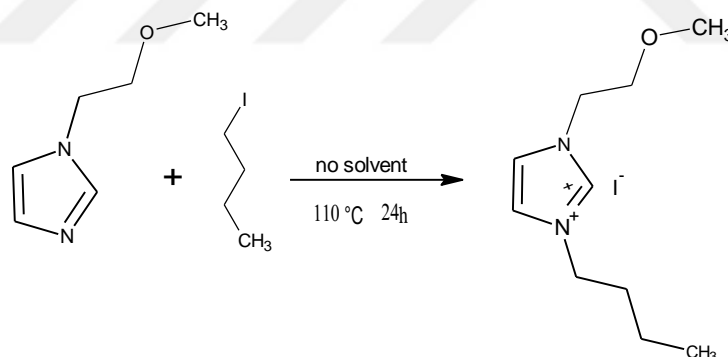


Figure 3.5: Stirring 1-(2-methoxyethyl)-1H-imidazole and 1-Iodobutane.

1-(2-methoxyethyl)-1H-imidazole (3.00 g; 23.8mmol) and 1-Iodobutane (4.81g; 26.1mmol) stirred at 110 °C for 24hours (Figure 3.5). Product was washed with diethylether (10mL) and recrystallized from dichloromethane/diethylether. The solvent was evaporated under vacuum and dark brown oil product was obtained. Yield: % 88. ¹H-NMR and FT-IR spectrums are given below in Figure 3.6 and Figure 3.7 respectively.

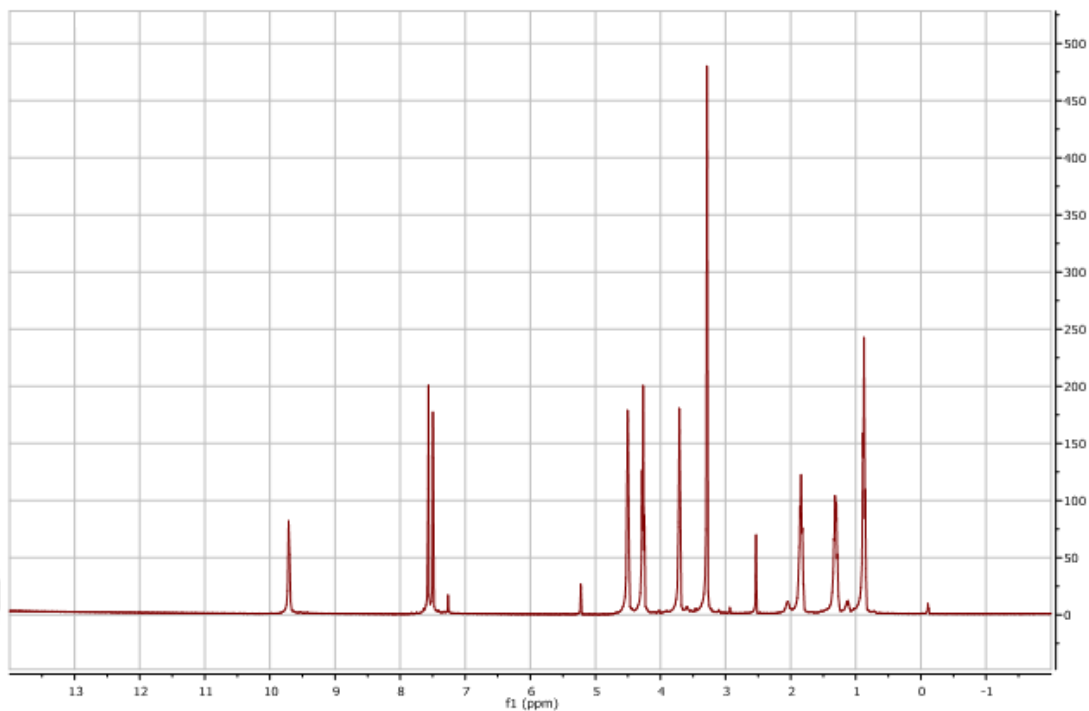


Figure 3.6: ¹H-NMR spectrum of MY2.

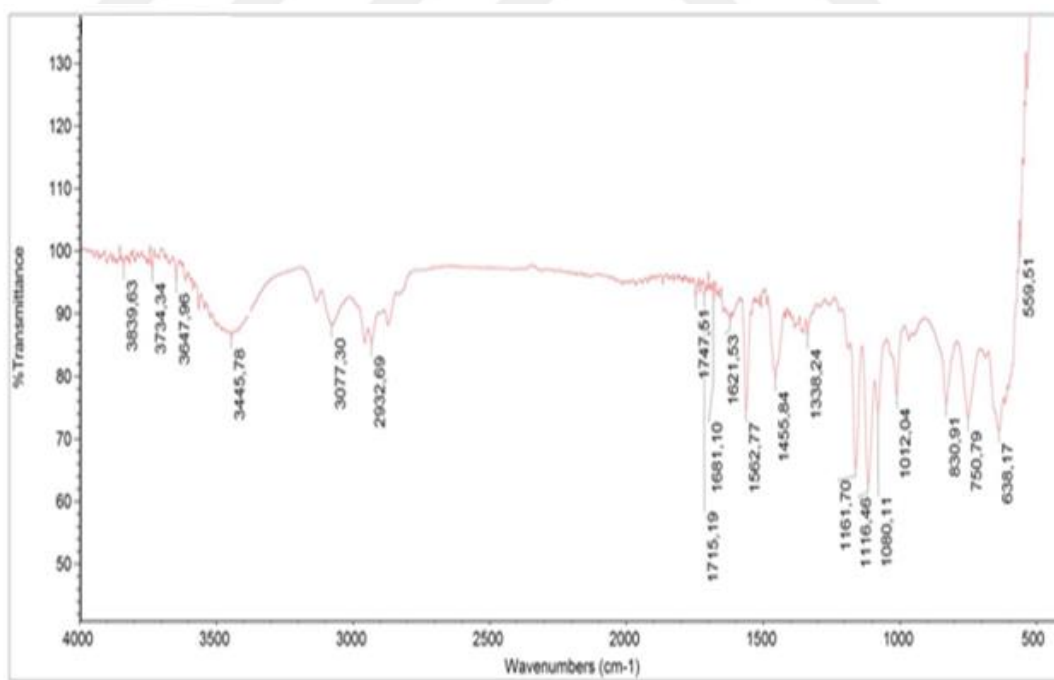


Figure 3.7: FT-IR spectrum of MY2.

3.2.3 3-butyl-1-propyl-1*H*-imidazol-3-ium iodide (MY1)



Figure 3.8: Stirring Imidazole and KOH in DMSO.

Imidazole (1.51g; 22.2mmol) and KOH (1.86g; 33.3mmol) were dissolved in DMSO (10mL) and stirred at room temperature for 5 hours. 1-Bromopropane (3.00g; 24.4mmol) was added. The mixture was stirred at 50 °C for 48 hours (Figure 3.8). After then, the reaction mixture was diluted with water (500mL) and extracted with CHCl₃ (5x25mL). The solvent was separated from the organic fractions affording the product. The solvent was removed and the product washed with hexane and diethylether. After, solvents were evaporated under vacuum and orange oil formed.

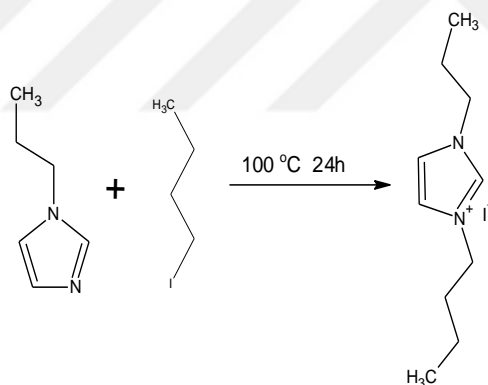


Figure 3.9: Stirring 1-propyl-1*H*-imidazole and 1-Iodobutane.

1-propyl-1*H*-imidazole (1.26g; 11.4mmol) and 1-Iodobutane (2.32g; 12.6mmol) stirred at 110 °C for 24 hours (Figure 3.9). Product was washed with diethylether (10mL) and recrystallized from dichloromethane/diethylether. The solvent was evaporated under vacuum and dark brown oil was obtained. Yield: % 80. ¹H-NMR and FT-IR spectrums are given below in Figure 3.10 and Figure 3.11 respectively.

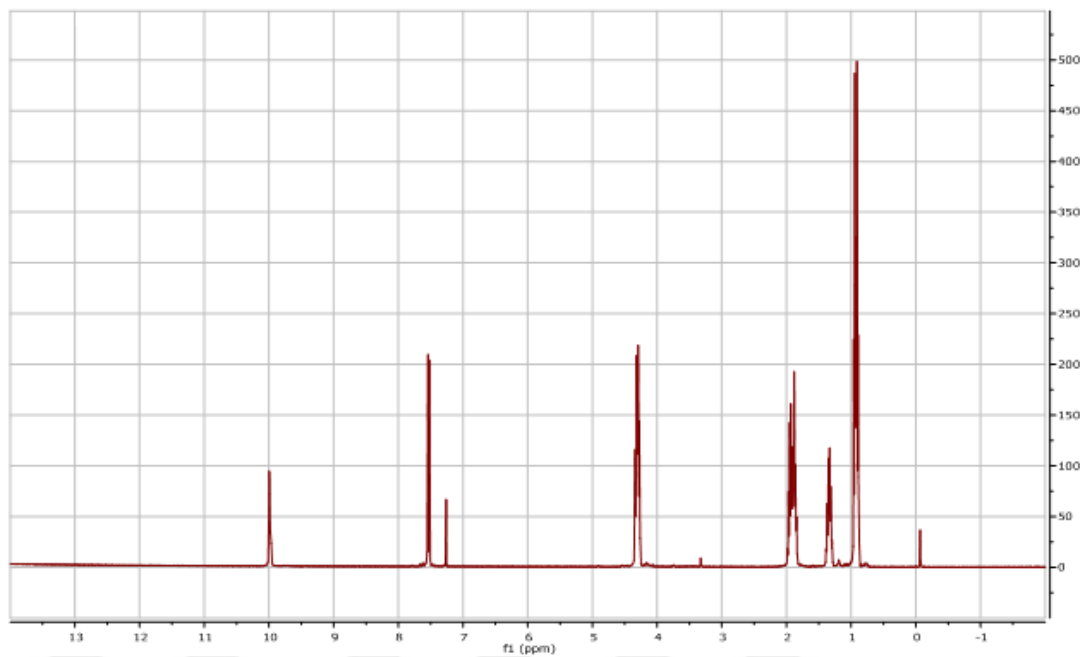


Figure 3.10: ¹H-NMR spectrum of MY1.

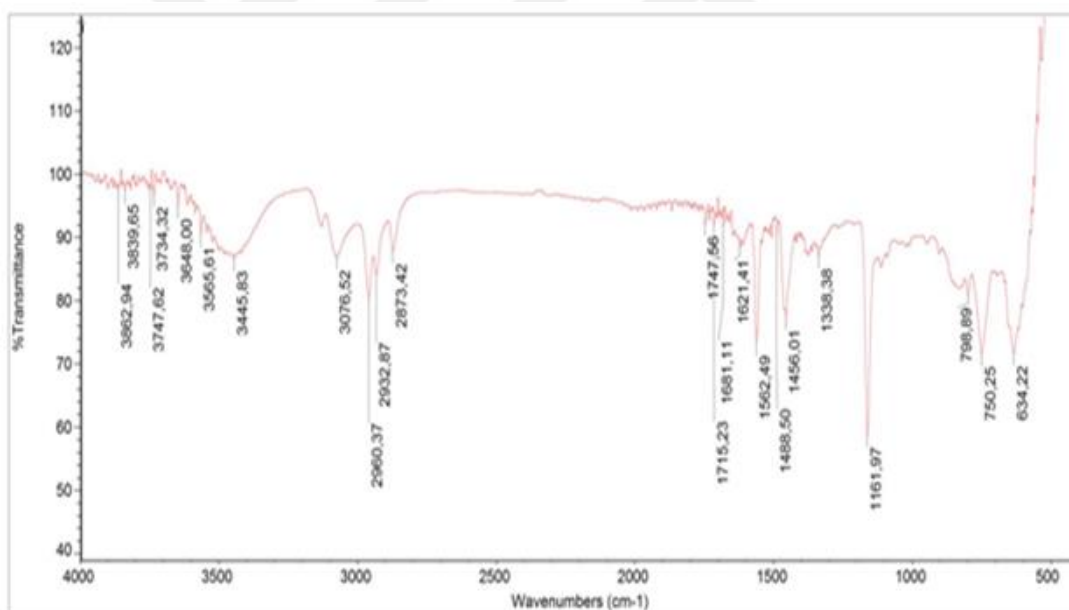


Figure 3.11: FT-IR spectrum of MY1.

3.3 Electrochemical Properties

Electrochemical studies were performed using CH Instruments CHI440B model cyclic voltammetry instrument. Three electrode cells were used for the study. Glassy

carbon was used as the working electrode and platinum wire as the auxiliary electrode, Ag wire as the reference electrode and 0,1 M TBAPF6 (tetrabutylammonium hexafluorophosphate) in the acetonitrile solution as the supporting electrolyte were used. Electrochemical studies were carried out in chloroform and DMSO solutions. Due to the low solubility of some of the synthesized compounds, the solute in DMSO was added into the electrolyte solution. The measurement is started after it has been seen that there is no peak in the potential range to be worked on before each scan. Also, before each measurement, Ar gas was passed through the sample for 10 minutes to remove oxygen from the environment. The scanning process was performed at speeds between 0,1-0,5 V / s [23,13].

Utilizing the CV voltammograms shown below, E_{HOMO} values of the ionic liquids were calculated. HOMO-LUMO values of an organic compound can be found by this equation. One of the important points to be aware of when values are found is that the initial oxidation and reduction peaks are taken from the graphs. Because the peaks of the initial oxidation or reduction are the peaks of the compound in a neutral state. Subsequent peaks or peaks may belong to anions or cations. In this case, the actual HOMO-LUMO value of the compound cannot be found. The following equation has been used in the calculation of HOMO-LUMO values [34].

$$E_{\text{HOMO}}: -(E_{\text{ox}}+4.4)\text{eV} \quad (1)$$

$$E_{\text{LUMO}}: -(E_{\text{red}}+4.4)\text{eV} \quad (2)$$

The electrochemical properties of ionic liquids were investigated by cyclic Voltammetry (CV) (Figure 3.12).

When analyzing the ionic liquids, it was determined that the oxidation potential of MY1(0.9 V) was lower than that of MY2(0.98 V) and standard ionic(1.1 V) liquid. Looking at the drawn energy diagram of these ionic liquids, it is seen that MY1 is the most effective in the reduction of the oxidized dye (Figure 3.13). Since other ionic liquids are very close to the HOMO energy level of the dye, electrons stimulated by back-reaction methods are thought to return to MY2 and the standard rather than

passing into the conduction band of TiO₂. For this study, a device called Transient Absorption Spectrometer (TAS) was used. MY1, which is synthesized as redox couple, can be said to be an effective ionic liquid in electrochemical dye-sensitive solar cells.

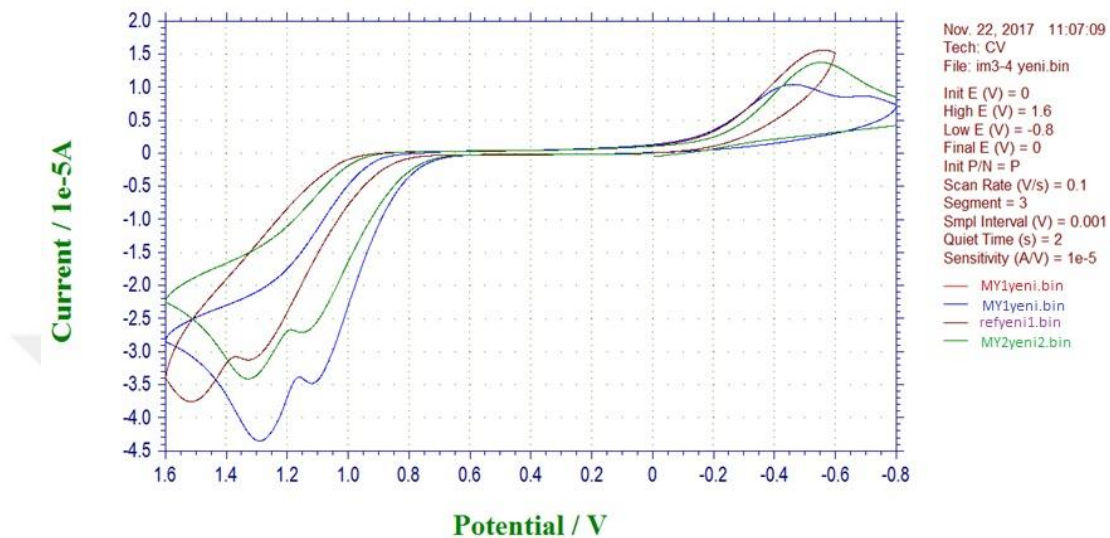


Figure 3.12: C-V graph.

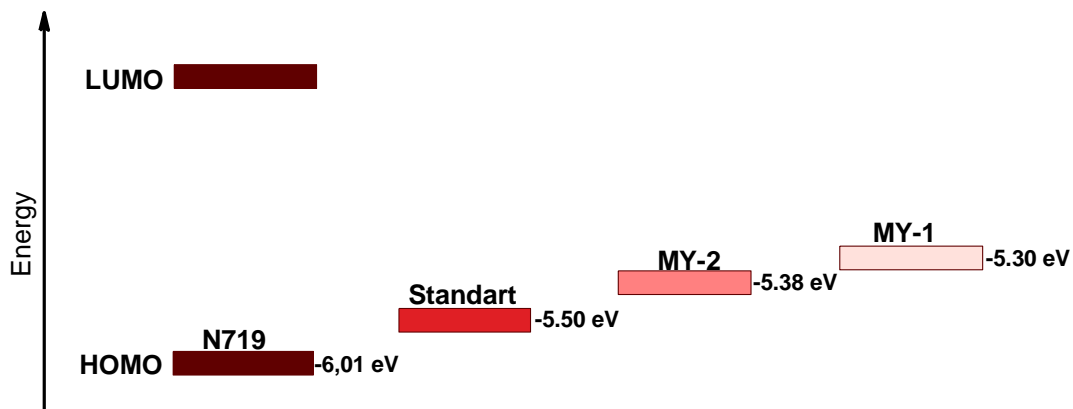


Figure 3.13: HOMO-LUMO Energy graph.

3.4 Spectroscopic Measurements of Synthesized Compounds

UV spectrophotometer was used to determine the photophysical properties of the synthesized compounds. Although iodine is used in these compounds and the colours of the prepared solutions are red, they are not capable of effective absorption. These ionic liquids are known as redox couple. These ionic liquids generally make absorption in the UV region. From the absorption graphs in Figure 3.14 it is observed that the maximum absorption of the synthesized compounds is under the visible region. When these ionic liquids are under the visible region, they do not absorb any light. This allows the incoming rays to be used more effectively. This also causes the dye used in the DSSC to absorb more light and increase the efficiency.

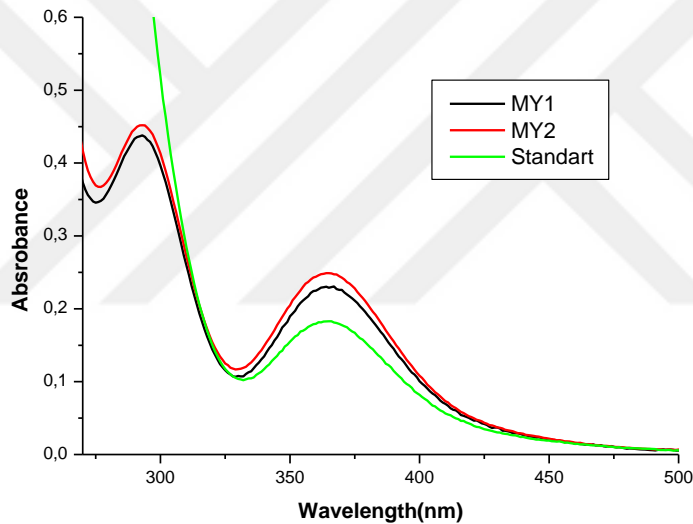


Figure 3.14: Absorbance-Wavelength Graph.

3.5 Current-Voltage Characteristics

- **Efficiency(η):**

Efficiency (η): The efficiency of the battery is expressed by the performance of the solar battery, and obtained by the ratio of the maximum power (P_{mpp}) and the light intensity coming to the solar cell surface (P_{sp}).

$$\eta = \frac{P_{mpp}}{P_{light}} = \frac{I_{sc} \cdot V_{oc} \cdot FF}{P_{light}} \quad (3)$$

- **Short Circuit Current (I_{sc}):**

When the voltage applied to the battery is $V_a = 0$ V, the measured current is the short circuit current (I_{sc}).

- **Open Circuit Voltage (V_{oc}):**

It is the measured voltage value when no current flows through the circuit. ($I = 0$)

- **The Current Density (J):**

It is a measure of the density in the electric circuit. It is referred to as a vector and is the ratio of the electric current to the cross sectional area. In SI the current density can be expressed in amperes per square meter or coulomb / second / square meter.

- **Maximum Power Point (MPP):**

The power obtained when the solar battery voltage V_a is applied is defined as the product of the current generated in the applied potential and the potential (V_a). The point where the highest power is obtained is called the Maximum Power Point (MPP), and the current and voltage at this point are expressed as the maximum current of the solar battery (I_{mpp}) and the maximum voltage (V_{mpp}).

$$P = V_a \cdot I \text{ [mW]} \quad (4)$$

$$P_{mpp} = V_{mpp} \cdot I_{mpp} \text{ [mW]} \quad (5)$$

- **Filling Factor (FF):**

Filling factor is a measure of the quality of the battery as a power source and is the ratio of the maximum power to the multiplication of short circuit current and open circuit voltage.

$$FF = \frac{V_{mpp} \cdot I_{mpp}}{V_{oc} \cdot I_{sc}} = \frac{P_{mpp}}{V_{oc} \cdot I_{sc}} \quad (6)$$

3.6 Photovoltaic Performance

3.6.1 I-V Graphs (Current-voltage (J-V) Characterization

Photovoltaic systems are generally characterized by measuring the varying current density, which depends on the applied tension in different radiation intensities, including darkness. At the same time, the photon under monochromatic light with low radiation intensity is also known as the incident photon to current conversion efficiency (IPCE) is a technique that is very often measured in systems and gives very important information about the operation of the system. In this thesis, we will focus on the design and synthesis of organic paints used mainly in solar batteries, but it is worthwhile to keep some general information in mind.

The J-V characteristics of DSSCs were obtained by using the Keithley 2400 Source-Meter Unit and Labview software. The measurements were made a $100\text{mW}/\text{cm}^2$ AM1.5 light intensity by solar simulator (KHS with 750 W Xe lamp). QE-R E Enlitech was used to measure the incident photon to current conversion efficiency (IPCE).

The active electrode area of DSSCs was 0.36 cm^2 .

Under standard global AM1.5 solar irradiation, the results of photovoltaic performance were summarized in “Table 3.1” and the J-V curves of DSSCs shown in “Fig 3.15” shows a short circuit current density (J_{sc}) of 12.79 mAcm^{-2} , open circuit voltage (V_{oc}) of 740 mV, fill factor of 0.71, and conversion efficiency 6.73% for MY1 DSSC. MY2 cell allow J_{sc} : 9.24 mAcm^{-2} , V_{oc} : 720 mV, FF: 0.64, η :4.25%. At same condition ref cell gave a J_{sc} : 6.27 mAcm^{-2} , V_{oc} : 660 mV, FF: 0.67, η :2.78%.

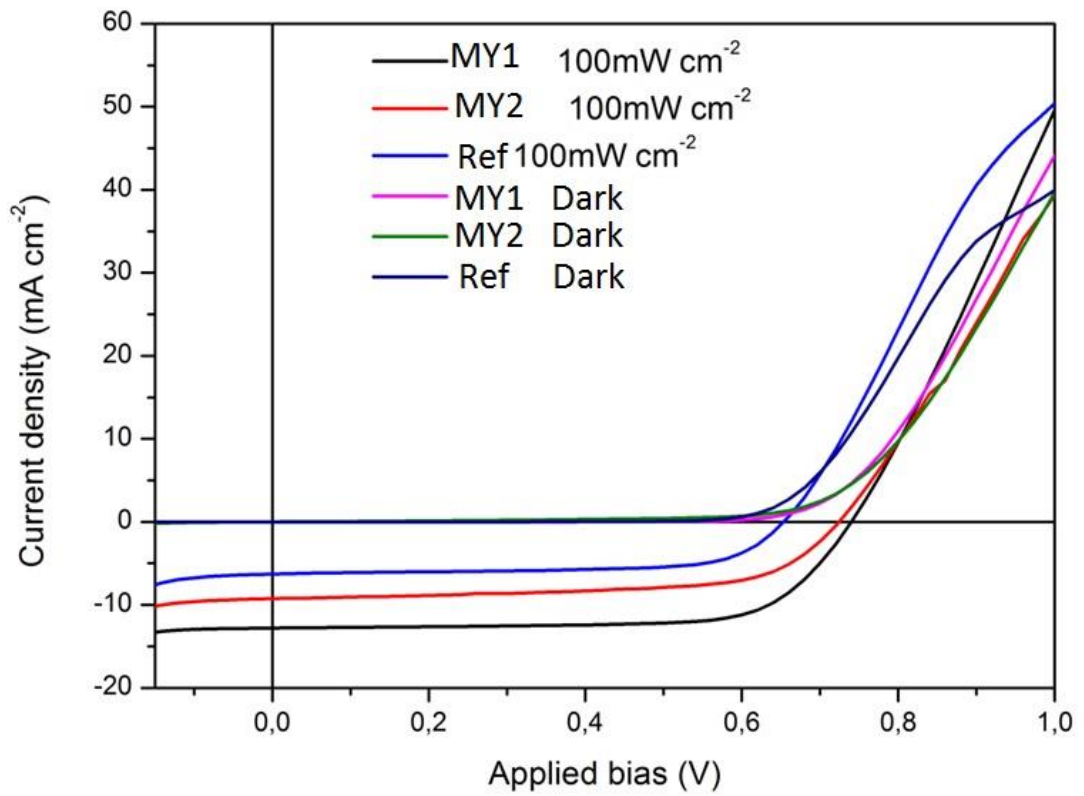


Figure 3.15: I–V curves of cells, under the dark and standard conditions (1.5AM, 100mW/cm²).

Table 3.1: Cell results.

	J_{sc} (mAcm⁻²)	V_{oc} (mV)	FF	η (%)
MY1	12.79	740	0.71	6.73
MY2	9.24	720	0.64	4.25
ref cell	6.27	660	0.67	2.78

As seen from the results, the higher J_{sc} value for the DSSC sensitized with MY1 is consistent with its larger value of IPCE. These results confirm that charge generation and charge injection yields are higher with the MY1 ionic liquid.

3.6.2 Incident photon-to-current conversion efficiency (IPCE)

The Incident Photon-to-Current Conversion Efficiency (IPCE), also called as External Quantum Efficiency (EQE), is a basic parameter used to characterize the solar cell performance in incident wavelengths.

$$\text{IPCE} = \frac{N_{\text{electron}}}{N_{\text{photon}}} = \frac{q \frac{N_{\text{electron}}}{s}}{q \frac{N_{\text{photon}}}{s}} = \frac{I_{\text{ph}}[\text{amp}]}{q \frac{N_{\text{photon}}}{s}} \quad (7)$$

IPCE is the product of three components; the light harvesting efficiency(LHE(λ)), the quantum yield(ϕ_{inj}) and the electron collection efficiency(η_{coll}).

IPCE shows the efficiency of conversion of light into electrical energy at a specific wavelength. It can also be described as photon to current efficiency.

Table 3.16 shows incident photon to current conversion efficiencies (IPCE) for MY1, MY2 and Ref cells. 520 nm is the maximum value of IPCE spectra, the efficiencies of MY1, MY2 and Ref cells about 43.51% , 26.92% and 18.62% respectively (Table 3.2).

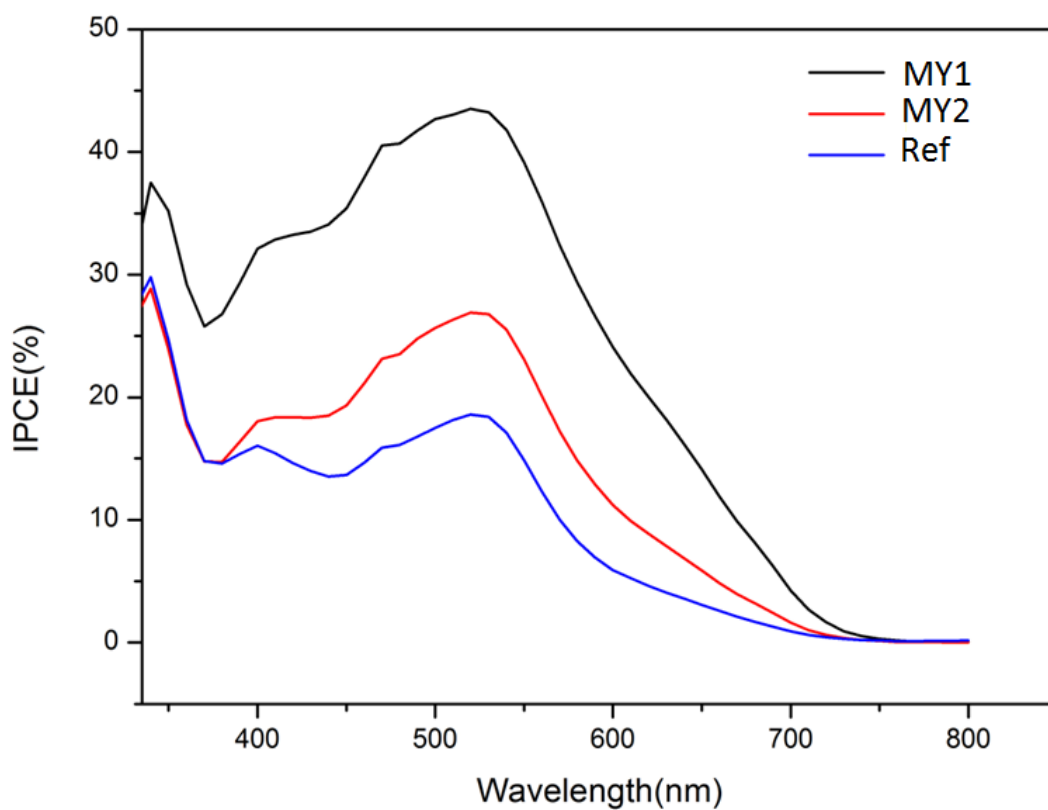


Figure 3.16: Incident Photon-to-Current Conversion Efficiency(IPCE) spectra of DSSCs.

Table 3.2: Cell efficiencies.

	η (%)
MY1	43.51
MY2	26.92
Ref	18.62

When the oxidation potential of ionic liquids is examined, it has been found that MY1 ionic liquid is oxidized more easily than other ionic liquids. This causes both the easier regeneration of the dye and the easier reception of the electrons it gives. It

is thought that the efficient use of load transfer in this way leads to both high efficiency and high IPCE.

3.6.3 Impedance

Resistance is the ability of a circuit resisting against the current. It is defined as the ratio between voltage and current. But this is the ideal resistor. In real world, there are much more complex circuit behaviors. AC current and voltage signals can be in different frequencies. Ideal resistor does not give us data about frequency so we need to use an impedance to obtain a more general data about the circuit [35].

$$R = \frac{E}{I} \quad (8)$$

$$Z = \frac{E_t}{I_t} \quad (9)$$

Electrical impedance spectroscopy (EIS) has been used to examine the charge transfer resistances (R_{ct}) of TiO_2 /dye/electrolyte interface. Many parameters can be obtained from electrochemical impedance spectroscopy, which is useful for investigating the interface charge transfer processes in DSSCs. Series resistance (R_s), charge transfer resistance (R_{ct}), capacitance of double layers (C_{dl}), diffusion coefficients (D) and life times of electrons (τ_e) can be found by this method. Generally, three semi-circles in Nyquist plot are seen in DSSCs (Figure 3.17). Double layer of electrolyte/ TCO electrodes gives the high frequency semi-circle by charge transfer resistance. Diffusion resistance of the redox species of the Warburg impedance electrolyte gives us the low frequency semi-circle. And the middle semi-circle is related to the charge recombination resistance (R_{rec}) of TiO_2 /Dye/Electrolyte interface.

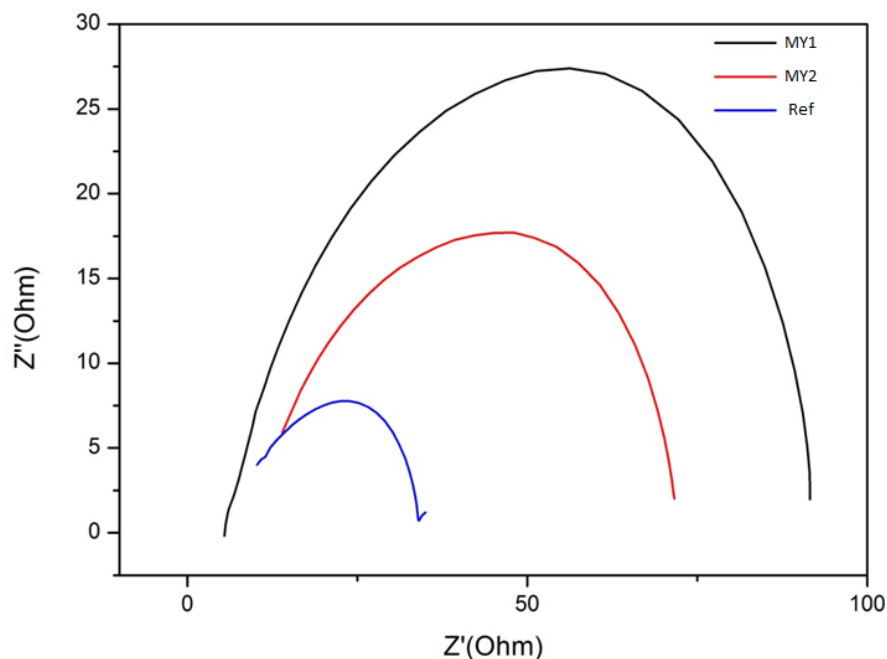


Figure 3.17: Nyquist plots of MY1, MY2 and Ref cells.

In this study, electrochemical characterization of dye-sensitized solar cells using ionic liquid was studied and it was compared with electrolytic cell which contains solvent. Electrochemical Impedance Spectroscopy (EIS) is a useful technique for identifying important battery parameters such as diffusion parameters for dye sensitized solar cells, chemical capacitance and recombination resistance. The volatility of the solvents is a major problem for long term tool performance. Solid or non-volatile phase electrolytes are alternative for electrolytes containing organic solvents. Room Temperature Ionic Liquids (RTIL) have high thermal stability and high ionic conductivity. These properties make ionic liquids advantageous for dye sensitizing solar cell applications.

It has been observed that the R_{ct} recombination resistance decreases due to the change in the length of the alkyl chain of the ionic liquid used according to the fit results obtained using electrochemical impedance spectroscopy. While the I_3^- concentration remains constant due to the ionic liquid change, the diffusion coefficient of De increases due to the change of diffusion rate and change of the

cation size, and as the electron injection from the electrolyte takes place very fast, the recombination decreases and the battery efficiency changes.



4. CONCLUSIONS

Our goal was to show an alternative synthesis of iodine, and we have found that the new ionic liquids are an alternative to the iodide redox couple.

In this thesis, different ionic liquids were synthesized which are used in the preparation of DSSC cells. These ionic liquids were used as redox couple in DSSC. The chemical structures of the synthesized compounds were elucidated by $^1\text{H-NMR}$ and FT-IR spectral data. According to the results of these spectra, it was determined that the compounds were obtained in a pure form.

Electrochemical properties of the compounds were determined using the CV method. Only the HOMO energy levels of the molecules were calculated by the CV measurements made. HOMO energy levels were found to vary between 5.30 and 5.50 eV.

The spectral properties of these synthesized ionic liquids were examined by using UV-Vis spectrophotometer. Absorption measurements revealed that all compounds absorbed in the UV region (**between 260 and 420 nm**). It has been observed that the two best absorbing MY-1 and MY-2 ionic liquids gave rise to two absorptive pikes, whereas the standard ionic liquid gives rise to a single absorptive pike. These ionic liquids did not seem to affect the absorption of the dye used.

Photovoltaic performances of DSSC cells which the synthesized compounds were used as photosensors were determined using I-V measurements. The highest yield was obtained with the MY-1 compound while the lowest yield was obtained with the ionic liquid used as standard. Looking at the IPCE spectra, it was seen that the conversion efficiency of MY-1 is better. Looking at the impedance spectrum, the MY-1 ionic liquid, which has the least resistance, appears to have better values.

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