





# DEVELOPMENT OF CARBOXYMETHYL CELLULOSE (CMC) BASED ELECTRO ACTIVE POLYMER NANO COMPOSITE & ITS POTENTIAL APPLICATION AS SOLID STATE DYE SENSITIZED SOLAR CELL

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# Mechanical & Chemical Engineering

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Organic solar cell research has developed during the past 30 years, but especially in the last decade it has attracted scientific and economic interest triggered by a rapid increase in power conversion efficiencies. This revolution was achieved by the introduction of new techniques, new and improved materials, engineering skills and more sophisticated device structures. Today, solar power conversion efficiencies in excess of 4% have been accomplished with several device concepts. Though efficiencies of these thin-film and other organic devices have not yet reached those of their inorganic counterparts ( $\approx 10-20\%$ ). The perspective of cheap production drives the development of organic photovoltaic devices further in a dynamic way. There are two important production techniques that are usually used either wet solution processing or dry thermal evaporation of the organic constituents. Now days, a lot of research in dye sensitized solar cell are going on all over the world. The field of organic solar cells profited well from the development of light-emitting diodes based on similar technologies, which have entered the market recently. We have tried to design and fabricate a solar cell with titanium dioxide (TiO<sub>2</sub> as an electron acceptor) and natural dye (red amaranth

as low cost electron donor) incorporated with carboxymethyl celluoise (CMC) Nano composite film. Thus, we have shown and compare all the electrical properties (current-voltage, resistance-conductivity, and voltage-resistance), mechanical properties (Hardness, tensile strength, elongation at break), optical properties (UV-visible spectroscopy) and morphological properties (top & rear side of the Nano composite film).

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### **CHAPTER ONE**

#### **1.0 INTRODUCTION**

#### **1.1 General Background**

Now days, Polymer photovoltaic devices offer great technological potential as a renewable, alternative source of electrical energy. The demand for inexpensive renewable energy sources is the driving force behind new approaches in the development of low-cost photovoltaic devices. In the last couple of years, increased effort has been put into the development of solar cells based on organic molecules and conjugated polymers.

Significant progress has been achieved in the synthesis of various types of polymer-Nano composites and in the understanding of the basic principles regarding optical, electronic and magnetic properties over the last few years. As a result Nano composite-based device such as photovoltaic solar cell has been developed, often using chemically orientated synthetic methods such as soft lithography, lamination, spin-coating or solution casting [1].

Organic, molecules are not generally known as good conductors, but there is nonetheless great interest in their electronic properties. Their flexibility in design and compatibility with a wide range of substrates and cost-effectiveness make them appealing for use in low-cost, flexible solar cells. One way to make organic material more conductive is to add impurity atoms that donate mobile charges to their molecular host [2].

Even though all the polymers are not electrically conductive, however most of them shear some intrinsic electrical properties. These properties are main subject of interest in organic electronics. A few research works have been done on natural polymer based organic electronic devices. In this study we have an exciting objective of investigating the electrical properties of naturally available polymers.

Carboxymethyl cellulose (CMC) or cellulose gum is a cellulose derivative with carboxymethyl groups (-CH<sub>2</sub>-COOH) bound to some of the hydroxyl groups of the

glucopyranose monomers that make up the cellulose backbone. It is often used as its sodium salt, sodium carboxymethyl cellulose. It is synthesized by the alkali-catalyzed reaction of cellulose with chloroacetic acid. The polar (organic acid) carboxyl groups render the cellulose soluble and chemically reactive.

The functional properties of CMC depend on the degree of substitution of the cellulose structure (i.e., how many of the hydroxyl groups have taken part in the substitution reaction), as well as the chain length of the cellulose backbone structure and the degree of clustering of the carboxymethyl substituents [3].

Titanium dioxide (TiO<sub>2</sub>), also known as titanium (IV) oxide or titania, is the naturally occurring oxide of titanium. It has been widely used as a photo catalyst for solar energy conversion and environmental applications because of its low toxicity, abundance, high photo stability, and high efficiency [4]. However, the application of pure TiO<sub>2</sub> is limited, because it requires ultraviolet (UV) light, which makes up only a small fraction ( < 4%) of the total solar spectrum reaching the surface of the earth. Therefore, over the past few years, considerable efforts have been directed towards the improvement of the photo catalytic efficiency of TiO<sub>2</sub> in the visible (vis)-light region. [5–7].

Green plants and their fruits and flowers are a fantastic source of natural dyes that absorb visible light. The red pigments found in raspberries or blackberries can work especially well in Dye Solar Cells. Red fruits are very handy for experimenting in training courses where the use of synthetic dyes can be an issue. The sensitization of titanium dioxide by natural dyes consists of soaking the titania electrode in mashed fruits. Complete staining can take from several minutes to several hours, while the dye molecules from the fruit juice naturally adsorb onto the titania particles. The longer the electrode soaks in the dye, the better dyed the titania will be [8 -12].

The results presented in this work indicated that it is possible to enhance the mechanical properties and electrical property of the Nano composite film which may be used in the field of organic semiconductor and organic photovoltaic solar cells in the present days.

#### **1.2 Review of Some Earlier Work**

During the last few years, many researchers have studied the various properties (Physical, mechanical, electrical, thermal and structural etc.) of conjugated Nano composites film and solar cells that were prepared for solar cell application. Considerable amounts of research work have done in different parts of the world. In this section a survey on the reported works on conjugated polymer based solar cell is given.

Franz Padinger et al. [13] have improved efficiencies of organic solar cells based on an interpenetrating network of a conjugated polymer and a fullerene which is available polymer as donor and acceptor materials for commercial use. They have developed a postproduction treatment that improves the performance of solar cells based on poly (3-hexylthiophene) (P3HT) and [6,6]-phenyl C<sub>61</sub>-butyric acid methyl ester (PCBM) by means of a tempering cycle at elevated temperatures in which an external voltage is simultaneously applied, resulting in a significant increase of the short-circuit current. Using this postproduction treatment, an enhancement of the short-circuit current density,  $I_{sc}$ , to 8.5 mA cm<sup>-2</sup> under illumination with white light at an illumination intensity of 800 W m<sup>-2</sup> and an increase in

external quantum efficiency (IPCE, incident photon to collected electron efficiency) to 70 % was demonstrated.

Wendy U. Huynh et al. [14] have demonstrated that semiconductor nanorods can be used to fabricate readily processed and efficient hybrid solar cells together with polymers. By controlling nanorod length, the distance on which electrons are transported directly through the thin film device can be changed. Tuning the band gap by altering the nanorod radius they optimized the overlap between the absorption spectrum of the cell and the solar emission spectrum. A photovoltaic device consisting of 7-nanometer by 60-nanometer CdSe nanorods and the conjugated polymer poly-3 (hexylthiophene) was assembled from solution with an external quantum efficiency of over 54% and a monochromatic power conversion efficiency of 6.9% under 0.1 milli watt per square centimeter illumination at 515 nanometers. Under Air Mass (A.M.) 1.5 Global solar conditions, they obtained a power conversion efficiency of 1.7%.

Peng Wang et al. [15] have shown a Dye-sensitized Nano crystalline Solar Cells (DSC) with unprecedented stable performance under both thermal stress and soaking with light, matching

the durability criteria applied to silicon solar cells for outdoor applications. The cell uses the amphiphilic ruthenium sensitizer *cis*-RuLL' (SCN)<sub>2</sub> (L = 4,4'-dicarboxylic acid-2,2'-bipyridine, L'= 4,4'-dinonyl-2,2'-bipyridine) in conjunction with a quasi-solid-state polymer gel electrolyte, reaching an efficiency of >6% in full sunlight (air mass 1.5, 100 mW cm–2). A convenient and versatile new route is reported for the synthesis of the heteroleptic ruthenium complex, which plays a key role in achieving the high-temperature stability. Ultra microelectrode voltammetry measurements show that the triiodide/iodide couple can perform charge transport freely in the polymer gel. The cell sustained heating for 1,000 h at 80<sup>o</sup>C, maintaining 94% of its initial performance. The device also showed excellent stability under light soaking at 55<sup>o</sup>C for 1,000 h in a solar simulator (100 mW cm–2) equipped with a ultraviolet filter. The present findings should foster widespread practical application of dyesensitized solar cells

Michael W. Rowell et al. [16] fabricated flexible transparent conducting electrodes by printing films of single-walled carbon Nanotube (SWNT) networks on plastic and had demonstrated their use as transparent electrodes for efficient, flexible polymer-fullerene bulk-hetero-junction solar cells. The printing method produced relatively smooth, homogeneous films with a transmittance of 85% at 550 nm and a sheet resistance  $R_s$  of 200/cm. Cells were fabricated on the SWNT/plastic anodes identically to a process optimized for ITO/glass. Efficiencies, 2.5% AM1.5G, were close to ITO/glass and were affected primarily by  $R_s$ . Bending test comparisons with ITO/plastic show the SWNT/plastic electrodes to be far more flexible.

A. Breeze et al. [17] investigated polymer-based photovoltaic cells. They presented initial results on a novel energy conversion device that uses both semiconducting polymers and organic small molecules as photoactive layers, using the advantageous properties of both classes of materials. By incorporating polymers into the device, they saw that the results were

surprisingly good for a planar organic device with a notable increase in efficiency versus analogous devices constructed from only small molecule organics.

Ho Chang et al. [18] study employs chlorophyll extract from pomegranate leaf and anthocyanin extract from mulberry fruit as the natural dyes for a dye-sensitized solar cell

(DSSC). A self-developed Nano fluid synthesis system is employed to prepare TiO<sub>2</sub> Nano fluid with an average particle size of 25 nm. Electrophoresis deposition was performed to deposit TiO<sub>2</sub> nanoparticles on the indium tin oxide (ITO) conductive glass, forming a TiO<sub>2</sub> thin film with the thickness of 11  $\mu$ m. Furthermore, this TiO<sub>2</sub> thin film was sintered at 450 °C to enhance the thin film compactness. Sputtering was used to prepare counter electrode by depositing Pt thin film on FTO glass at a thickness of 20 nm. The electrodes, electrolyte, and dyes were assembled into a cell module and illuminated by a light source simulating AM 1.5 with a light strength of 100 mW/cm<sup>2</sup> to measure the photoelectric conversion efficiency of the prepared DSSCs. According to experimental results, the conversion efficiency of the DSSCs prepared by chlorophyll dyes from pomegranate leaf extract is 0.597%, with opencircuit voltage ( $V_{OC}$ ) of 0.56 V, short-circuit current density ( $J_{SC}$ ) of 2.05 mA/cm<sup>2</sup>, and fill

factor (FF) of 0.52. The conversion efficiency of the DSSCs prepared by anthocyanin dyes from mulberry extract is 0.548%, with  $V_{OC}$  of 0.555 V and  $J_{SC}$  of 1.89 mA/cm<sup>2</sup> and FF of 0.53. The conversion efficiency is 0.722% for chlorophyll and anthocyanin as the dye mixture, with  $V_{OC}$  of 0.53 V,  $J_{SC}$  of 2.8 mA/cm<sup>2</sup>, and FF of 0.49.

M. Hossein et al. [19] interpreted that dye sensitized solar cell (DSSC) fabrication procedure has been employed using natural pomegranate juice for sensitization of nanocrystalline TiO2. Platinum and graphite coated electrodes were prepared by pulse current electron deposition and soot staining method for use as counter electrodes. Photovoltaic parameters like short circuit current (ISC), open circuit voltage (VOC) and fill factor (FF) were evaluated for fabricated cells. Although the fill factor for both cells was found to be 45%, ISC and VOC for cells operating with carbon and platinum coated counter electrodes were increased from 360 to 400 mV and from 175 to 200  $\mu$ A respectively. Overall conversion efficiencies of fabricated

DSSC found to be 1.5 % for cell operated with platinum electrodeposited and 0.9 % for carbon coated counter electrodes.

#### **1.3 Objectives of the Present Research Work**

The work deals with to design the Nano composite film with titanium dioxide ( $TiO_2$ ) & natural dye (red amaranth) incorporated with carboxymethyl cellulose. Our primary attention is focused to the electrical, mechanical, optical and morphological properties of a Nano composite based on natural dye and  $TiO_2$  modified carboxymethyl cellulose films in the present study.

- Different electrical properties such as current-voltage, voltage-resistance, and conductivity-resistance and DC measurement of the composites have been investigated.
- In addition to electrical properties, mechanical properties (tensile strength, elongation at break), morphological analysis, UV-visible light absorption analysis have been observed as well.

These experimental results will help to find out the suitability of the Nano composite film for solar cell application and to fabricate the solar cell.

## CHAPTER TWO

### SOLAR CELLS & ITS TYPES

## **2.1 Introduction**

The word energy comes from the Greek ἐνέργεια - *energeia*, "activity, operation", from ἐνεργός - *energos*, "active, working"[20]

Energy is a quantity that can be assigned to every particle of the system and system of objects as it can maintain a consequence of the state of that matters, system and others.

The list of energy technologies excluded is an indicator of which problems the alternative technologies are intended to address. Controversies regarding dominant sources of energy and their alternatives have a long history. The nature of what were regarded alternative energy sources has changed considerably over time, and today, because of the variety of energy choices and differing goals of their advocates, defining some energy types as "alternative" which is highly controversial [21].

There are several forms of energy. The forms of energy are often named after a related force. German physicist Hermann von Helmholtz established that all forms of energy are equivalent - energy in one form can disappear but the same amount of energy will appear in another form [22-23].

Now days, the demand for making solar cell is increasing day by day. Because the conventional power energy is decreasing day by day. People all over the world are trying to use the energy that has been produced from the nature.

## 2.2 Solar Energy

Sun is the source of energies. Prehistoric plants stored the Sun's energy in their leaves, and when they died and eventually formed coal seams, that energy was still there. So when we burn coal or any fossil fuel, we are releasing chemical energy that was stored in plants millions of years ago. The same goes for wind and wave power. Waves occur because of winds, and winds blow because the Sun warms our atmosphere. Warm air tends to rise, and

winds are due to other air moving in to replace it. Solar energy is renewable energy is sustainable in its production; the available supply will not be diminished for the foreseeable future - millions or billions of years and have no direct waste products. However the sun provides approximately 100000 terawatts to the earth which is about 10000 times more than the present rate of the world's present energy consumption [24]. In the Phoenix, Arizona area, for example, the average annual solar radiation is 5.7 kWh/m<sup>2</sup>/day [25] or 2080.5 kWh/m<sup>2</sup>/year. Electricity demand in the continental U.S. is  $3.7 \times 10^{12}$  kWh per year. Photovoltaic cells are being increasingly used to tap into the huge resource and will play a key role in future sustainable energy system.

### 2.3 Solar Cells

The term "photovoltaic" comes from the Greek  $\varphi \tilde{\omega} \zeta (ph \bar{o}s)$  meaning "light", and from "Volt", the unit of electro-motive force, the volt, which in turn comes from the last name of the Italian physicist Alessandro Volta, inventor of the battery (electrochemical cell). The term "photo-voltaic" has been in use in English since 1849 [26, 27].

*Photovoltaic* is the field of technology and research related to the practical application of photovoltaic cells in producing electricity from light, though it is often used specifically to refer to the generation of electricity from sunlight. Cells can be described as *photovoltaic* even when the light source is not necessarily sunlight (lamplight, artificial light, etc.). In such cases the cell is sometimes used as a photo detector (for example infrared detectors), detecting light or other electromagnetic radiation near the visible range, or measuring light intensity.

The operation of a photovoltaic (PV) cell requires 3 basic attributes:

- (1) The absorption of light, generating either electron-hole pairs or exactions
- (2) The separation of various types of charge carriers
- (3) The separate extraction of those carriers to an external circuit

In contrast, a solar thermal collector collects heat by absorbing sunlight, for the purpose of either direct heating or indirect electrical power generation. "Photo electrolytic cell" (photo electrochemical), on the other hand, refers either a type of photovoltaic cell (like that

developed by A.E. Becquerel and modern dye-sensitized solar cells) or a device that splits water directly into hydrogen and oxygen using only solar illumination.

## **2.4 Solar Cell Configuration**

Solar cells are often electrically connected and encapsulated as a module. Photovoltaic modules often have a sheet of glass on the sun up side, allowing light to pass while protecting the semiconductor wafers from the elements (rain, hail, etc.). Modules are then interconnected, in series or parallel, or both, to create an array with the desired peak DC voltage and current.

To make practical use of the solar-generated energy, the electricity is most often fed into the electricity grid using inverters; in stand-alone systems, batteries are used to store the energy that is not needed immediately. To deliver the desired amount of energy, the solar cells can be combined in series and parallel circuits. Such a design is called a solar panel. Further, the cell surface area can be increased, to allow stronger current from each cell.

## 2.5 Types of Solar Cells

In 1883, when Charles Fritts first made a selenium-based solar cell, he began a long chain of reproduction and evolution - a chain culminating thus far in the many types of solar cells we know today.

### 2.5.1 Carbon-based Solar Cells

Photovoltaic (PV) solar cells using carbon nanotube conductive coatings and circuits are underway to becoming one of the latest alternatives to silicon-based PV cells. Alternatives to the silicon wafer in PV cells are steadily being researched and monitored as the solar power industry continues to rise in quality, popularity, and practicality.

Carbon nanotube solar cells are under investigation for several purposes, including potential space applications. It is also believed that thin films of this material on conventional silicon solar cells can boost the overall efficiency [28].



Figure 2.1: Carbon based solar cell

### 2.5.2 3-D Solar Cells

Three-dimensional solar cells are yet another front in the quest for high efficiency, low-cost solar cells. 3-D cells can capture nearly all the light that strikes them, greatly boosting the efficiency of photovoltaic cells while reducing size, weight and complexity.

3-D cells are still very much in their infancy, but research is exciting and ongoing [29]



Figure 2.2: 3-D solar panel

#### **2.5.3 Foil Solar Cells**

With foil-based cells, copper and other materials replace silicon as the semiconductors and are placed into a protective metal foil. While traditional cells require heavy module packaging for protection, solar cells placed in flexible titanium or stainless steel foils are resistant and lightweight at the same time. The material is even thinner than household aluminum foil and comparably flexible, enabling the cells to bend into curved surfaces [30].



Figure 2.3: Foil solar cell

#### **2.5.4 Polymer Solar Cells**

Polymer solar cells are third generation solar cells. A solar cell is constructed when fullerenes, or molecules comprised solely of carbon, are injected into polymer. Then, as visible light moves from the polymer chain to a fullerene molecule, electrons are liberated from their macromolecular structure, producing an electrical charge. The process, however, is slow, and present polymer cell efficiency is at a low 5 percent [31].



Figure 2.4: Polymer solar cell

#### 2.5.5 Dye-Sensitized Solar Cells

In the late 1960s it was discovered that illuminated organic dyes can generate electricity at oxide electrodes in electrochemical cells [32]. In an effort to understand and simulate the primary processes in photosynthesis the phenomenon was studied at the University of California at Berkeley with chlorophyll extracted from spinach (bio-mimetic or bionic approach) [33]. On the basis of such experiments electric power generation via the dye sensitization solar cell (DSSC) principle was demonstrated and discussed in 1972 [34]. The instability of the dye solar cell was identified as a main challenge. Its efficiency could, during the following two decades, be improved by optimizing the porosity of the electrode prepared from fine oxide powder, but the instability remained a problem [35]. A modern DSSC, the Graetzel cell, is composed of a porous layer of titanium dioxide nanoparticles, covered with a molecular dye that absorbs sunlight, like the chlorophyll in green leaves. The titanium dioxide is immersed under an electrolyte solution, above which is a platinum-based catalyst. As in a conventional alkaline battery, an anode (the titanium dioxide) and a cathode (the platinum) are placed on either side of a liquid conductor (the electrolyte).

Sunlight passes through the transparent electrode into the dye layer where it can excite electrons that then flow into the titanium dioxide. The electrons flow toward the transparent electrode where they are collected for powering a load. After flowing through the external circuit, they are re-introduced into the cell on a metal electrode on the back, flowing into the electrolyte. The electrolyte then transports the electrons back to the dye molecules.

Dye-sensitized solar cells separate the two functions provided by silicon in a traditional cell design. Normally the silicon acts as both the source of photoelectrons, as well as providing the electric field to separate the charges and create a current. In the dye-sensitized solar cell, the bulk of the semiconductor is used solely for charge transport, the photoelectrons are provided from a separate photosensitive dye. Charge separation occurs at the surfaces between the dye, semiconductor and electrolyte.

The dye molecules are quite small (nanometer sized), so in order to capture a reasonable amount of the incoming light the layer of dye molecules needs to be made fairly thick, much thicker than the molecules themselves. To address this problem, a nanomaterial is used as a scaffold to hold large numbers of the dye molecules in a 3-D matrix, increasing the number of molecules for any given surface area of cell [36].

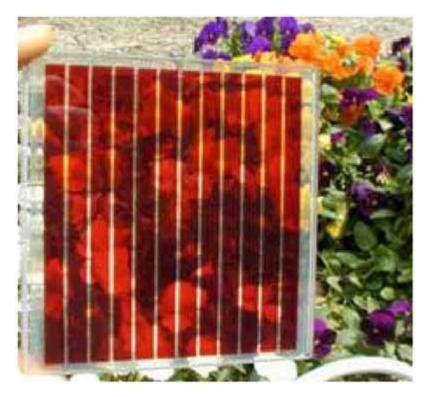


Figure 2.5: Dye sensitized solar cell

#### 2.5.6 Silicon Solar Cells

Solar grade, or bulk silicon, has a crystalline structure. Each silicon atom consists of three layers filled with electrons that increase in number toward the center of the atom. As electrons in the outer shell are released, the inner electrons move to take their place, resulting in a locked structure. This is why most silicon solar cells are either N-type or P-type silicon cells. Through a doping process, N-type cells have been chemically altered to include phosphorous, enabling more electrons to escape the outer shell of the atomic structure to become electricity. P-type silicon cells are doped with boron, which allows electrons to leave the atomic structure by creating positively charged holes in their place. The more free electrons are roaming, the higher the electrical charge [37].



Figure 2.6: silicon solar cell

### 2.5.7 Nano Crystal Solar Cells

Nano crystal, or quantum dot solar cells are part of what is called the 4th generation of solar cell technology. They are considered to be a part of the future of solar power but at this time are still deep in the testing phase. Nano crystal cells consist of a coating of Nano crystals on a silicon substrate.

New and promising modes of production include "spin-coating." It involves placing a specific amount of Nano crystal or quantum dot solution on a substrate, the substrate is then spun at a very fast rate and the solution subsequently spreads out to form an even coating [38].



Figure 2.7: Nano crystal solar cell

#### 2.5.8 Organic Photovoltaic Solar Cells

Organic photovoltaic cells are considered among the third generation of solar cells. Simply put, they use organic materials for light absorption and transporting electric charges. Potential advantages include easy, low-cost, and large-scale production as well as heightened flexibility. Organic molecules used in the construction of these solar cells are also flexible in that they can be chemically tuned to absorb a very wide spectrum of light in a relatively small material area. That plasticity should also allow for an incredibly high amount of applications. It is possible that organic solar cells could comprise liquids such as ink and be printed onto paper or other substrates at very small costs.

Organic solar cells have three types

- 1. **Single-layer** cells are the simplest, comprised of one organic PV material sandwiched between to metallic conductors.
- 2. **Multi-layer** organic solar cell, which contains two different layers of organic PV material carefully chosen to maximize the electrostatic forces created between the two.
- 3. The **dispersed hetero-junction** photovoltaic organic solar cell was developed. In this type those two layers, one an electron donor, one an electron acceptor, are mixed together to form a polymer. This enables a more efficient solar cell than its predecessors.

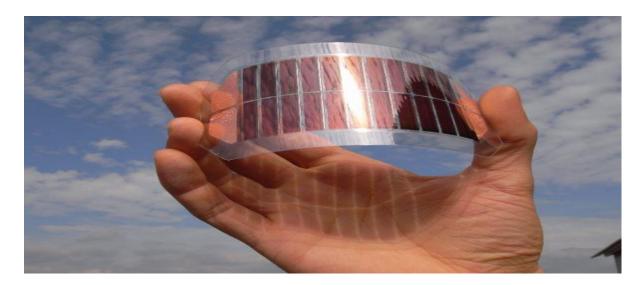


Figure 2.8: Organic photovoltaic solar cell

### 2.5.9 Advanced Thin-Film Solar Cells

Ironically, advanced thin-film solar cells are not that advanced, although the technology they inhabit is at the cutting edge of solar development. Potential products include solar paints, inks and spray-on solar cells that could spawn applications as revolutionary as solar windows, solar siding, solar clothing, solar cells that collect energy at night, and many, many others [39].

### 2.5.10 Thin-film Solar Cells

Thin-film solar cells are the next generation of solar cells

Thin-film solar cells are a rapidly growing category of solar photovoltaic cells that directly convert solar energy into electricity usable within the home. Thin-film cells consist of one or more layers of photovoltaic material on a substrate. Solar cells under the thin-film category are defined by the type of material used in cell construction.

Because of their slim size, thin-film solar cells have become the leader in Building Integrated Photovoltaic (BIPV). They can be easily incorporated into a building's design, typically as part of the roof structure in the form of solar shingles or rolls for metal roofing.

Thin-Film Types are several types.

Types of thin-film solar cells include:

- Amorphous silicon (aSi)
- Cadmium Telluride (CdTe)
- Copper indium gallium selenide (CIGS)
- Dye-sensitized solar cells (DSC or DSSC)
- Organic solar cells [40].

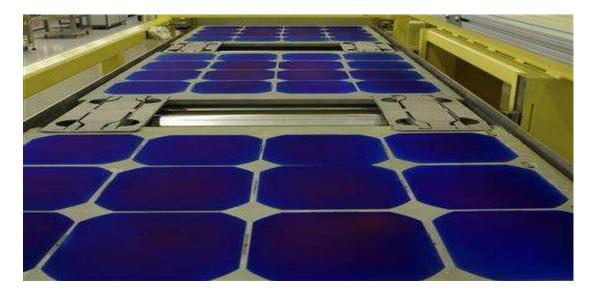


Figure 2.9: Thin solar cell

### 2.5.11 Carbon Nanotube Solar Cells

When light is shined onto the carbon nanotube, electrons are efficiently excited. These electrons are drawn down the tube by electrical contacts at either end. What makes nanotubes so special is that as the electrons flow down the nanotube, they get more excited and subsequently excite more electrons, which are uniformly "squeezed" out the end of the tube. These Cornell researchers have determined that carbon nanotubes may be the ideal photovoltaic cell because they allow excited electrons to create even more electrons, thus harnessing more of the photon energy from the light striking the tube [41].



Figure 2.10: Carbon Nano tube solar cell

#### 2.5.12 Hetero-junction and Multi-Junction Solar Cells

Multi-junction, or hetero-junction, solar cells are a type of thin-film solar cell. Most thin-film solar cells are inherently multi-junction. That's because a multi-junction cell consists of two or more thin layers of semiconductors, each designed to absorb a certain portion of the solar spectrum. The idea is that the combination of the semiconductors that form a multi-junction solar cell will absorb as close to the entire light spectrum as possible, thus creating maximum solar electricity from incident sunlight.

Multi-junction solar cells are playing a key role in the quest for high-efficiency solar cells. So far, thin-film solar cells that use hetero-junctions can absorb a vast amount of sunlight, but are relatively inefficient at converting that sunlight into usable electricity. CIGS solar cells are, however, continually showing improvement, having reached cell efficiencies near 20% and module efficiencies as high as 12.5%.

Thin-film hetero-junction solar cells are typically much cheaper to produce than conventional silicon-based solar cells. So, if researchers can match their absorption rates with higher efficiencies, these cells will be ready to take over the solar panel market [42].

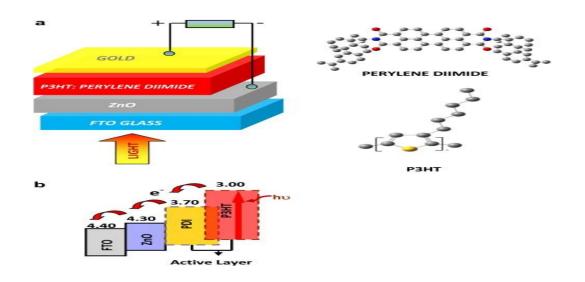


Figure 2.11: Bulk hetero-junction solar cell

#### 2.5.13 Hybrid Solar Cells

Hybrid solar cells combine organic and inorganic semiconductor materials in their cell structure. More specifically, organic polymers that absorb light and act as "electron donors" are combined with inorganic materials that act as "electron acceptors" and aid in electron transport as well. Hybrid solar cells are a type of third generation solar cell that offers a high potential for inexpensive, roll-to-roll manufacturing and high-efficiency power production.

Hybrid solar cells are multi-junction cells in that the two materials are placed in close contact with each other. Each layer acts as a junction through which photons of light must pass or be absorbed. This setup creates higher conversion efficiencies than a single material doing all the work on its own. The main challenges of hybrid solar cells, like other solar cells in development, include increasing conversion efficiency and limiting degradation over time [43].



Figure 2.12: Hybrid solar cell

#### 2.6 Needs for Organic Polymer Based Solar Cell

The widespread expansion in the use of inorganic solar cells remains limited due to the high costs imposed by fabrication procedures involving elevated temperature (400° to 1400°C), high vacuum, and numerous lithographic steps. Organic solar cells that use polymers which can be processed from solution have been investigated as a low-cost alternative with solar power efficiencies of up to 50% [44].

## CHAPTER THREE

## POLYMER, ORGANIC MATERIALS & NATURAL DYE

## **3.1 Introduction**

Polymer science is an important the subfield of materials science which deals with polymers, basically synthetic polymers such as plastics [45]. The field of polymer science includes researchers in multiple disciplines including chemistry, physics, and engineering.

## 3.2 Polymer

A polymer is a chemical compound or mixture of compounds consisting of repeating structural units created through a process of polymerization. Polymer is a large molecule composed of repeating structural units connected by covalent bonds. The word is derived from the Greek words  $\pi o \lambda v$  (poly), meaning "many"; and  $\mu \acute{e} po \varsigma$  (meros), meaning "part"[46]. Well known examples of polymers are plastics, DNA and proteins etc. A simple example is polypropylene whose repeating unit structure is shown in Fig. 3.1 & Fig. 3.2. Natural polymer materials such as shellac and amber have been in use for centuries. Biopolymers like proteins and nucleic acids play crucial roles in biological processes.

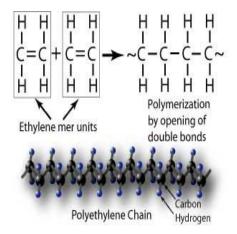


Figure 3.1: Structure of polymer

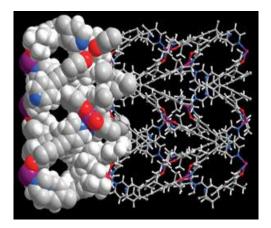


Figure 3.2: 3-D Structure of Polymer

### **3.3 Polymer Properties**

Polymer 'properties' can mainly divided into physical and chemical properties.

#### **3.3.1 Physical Properties**

#### a. Relationship between chain length and polymer properties

Increasing chain length tends to decrease chain mobility, increase strength and toughness, and increase the glass transition temperature (Tg). This is a result of the increase in chain interactions such as Van der Waals attractions and entanglements that come with increased chain length. These interactions tend to fix the individual chains more strongly in position and resist deformations and matrix breakup, both at higher stresses and higher temperatures. length Chain is related to melt viscosity roughly as 1:10 [47, 48 ], so that a tenfold increase in polymer chain length results in a viscosity increase of over 1000 times.

#### d. Young's Modulus of elasticity

Young's Modulus quantifies the elasticity of the polymer. It is defined, for small strains, as the ratio of rate of change of stress to strain. Like tensile strength this is highly relevant in polymer applications involving the physical properties of polymers, such as rubber bands.

#### i. Chain conformation

The space occupied by a polymer molecule is generally expressed in terms of radius of gyration, which is an average distance from the center of mass of the chain to the chain itself. Alternatively, it may be expressed in terms of pervaded volume, which is the volume of solution spanned by the polymer chain and scales with the cube of the radius of gyration [49].

Polymer has other notable properties like mixing behavior, branching, and chemical crosslinking.

#### c. Tensile strength

The tensile strength of a material quantifies how much stress the material will endure before failing [50, 51]. This is very important in applications that rely upon polymer's physical

strength or durability. For example, a rubber band with a higher tensile strength will hold a greater weight before snapping. In general, tensile strength increases with polymer chain length.

#### **b.** Crystallinity

Synthetic polymers may consist of both crystalline and amorphous regions; the degree of crystallinity may be expressed in terms of a weight fraction or volume fraction of crystalline material. Few synthetic polymers are entirely crystalline [52].

#### **3.3.2.** Chemical Properties of Polymers

The attractive forces between polymer chains play a large part in determining a polymer's properties. Because polymer chains are so long, these inter-chain forces are amplified far beyond the attractions between conventional molecules. Different side groups on the polymer can lend the polymer to ionic bonding or hydrogen bonding between its own chains. These stronger forces typically result in higher tensile strength and melting points.

The intermolecular forces in polymers can be affected by dipoles in the monomer units. Polymers containing amide or carbonyl groups can form hydrogen bonds between adjacent chains; the partially positively charged hydrogen atoms in N-H groups of one chain are strongly attracted to the partially negatively charged oxygen atoms in C=O groups on another. These strong hydrogen bonds, for example, result in the high tensile strength and melting point of polymers containing urethane or urea linkages. Polyesters have dipole-dipole bonding between the oxygen atoms in C=O groups and the hydrogen atoms in H-C groups. Dipole bonding is not as strong as hydrogen bonding, so polyester's melting point and strength are lower than Kevlar's (Twaron), but polyesters have greater flexibility.

## **3.4 ORGANIC SOLAR CELL**

An **organic solar cell** or **plastic solar cell** is a type of polymer solar cell that uses organic electronics, a branch of electronics that deals with conductive organic polymers or small organic molecules [53], for light absorption and charge transport to produce electricity from sunlight by the photovoltaic effect.

The plastic used in organic solar cells has low production costs in high volumes. Combined with the flexibility of organic molecules, organic solar cells are potentially cost-effective for photovoltaic applications. Molecular engineering (e.g. changing the length and functional group of polymers) can change the energy gap, which allows chemical change in these materials. The optical absorption coefficient of organic molecules is high, so a large amount of light can be absorbed with a small amount of materials. The main disadvantages associated with organic photovoltaic cells are low efficiency, low stability and low stability and low strength compared to inorganic photovoltaic cells.



Figure 3.3: Tandem Organic solar cell

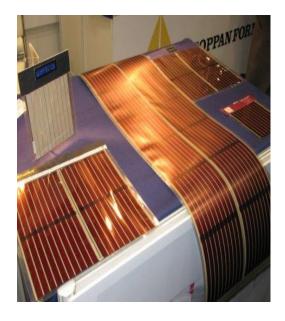


Figure 3.4: Thin film organic solar cell

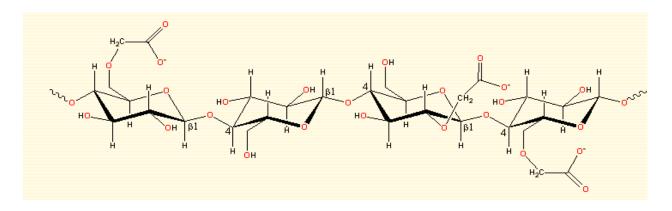


Figure 3.5: Flexible organic solar cell

## 3.5 Carboxymethyl Cellulose (CMC)

Carboxymethyl cellulose (CMC) or cellulose gum [53] is a cellulose derivative with carboxymethyl groups (-CH2-COOH) bound to some of the hydroxyl groups of the glucopyranose monomers that make up the cellulose backbone. It is often used as its sodium salt, sodium carboxymethyl cellulose [54]. CMC is a low viscosity technical grade dispersible Carboxymethyl Cellulose fluid loss additive designed to reduce API filtration rate with minimum increase in viscosity in water based drilling fluids.

### 3.5.1 Structural unit of CMC



**Figure 3.6:** The CMC structure is based on the  $\beta$ -(1 4)-D-glucopyranose polymer of cellulose. Different preparations may have different degrees of substitution, but it is generally in the range 0.6 - 0.95 derivatives per monomer unit [55].

#### **3.5.2 PREPARATION OF CONTENTS**

It is synthesized by the alkali-catalyzed reaction of cellulose with chloroacetic acid. The polar (organic acid) carboxyl groups render the cellulose soluble and chemically reactive.

The functional properties of CMC depend on the degree of substitution of the cellulose structure (i.e. how many of the hydroxyl groups have taken part in the substitution reaction), as well as the chain length of the cellulose backbone structure and the degree of clustering of the carboxymethyl substituents [54].



Figure 3.7: Carboxymethyl Cellulose (CMC)

#### 3.5.3 Advantages of CMC:

- Widely available and low cost polymer for fluid Loss control.
- It is an effective at providing filtration control in most water based drilling fluids.
- It is effective in low concentrations.
- It is Non–Toxic.
- It is not subjected to bacterial fermentation and calcium contamination.
- Can be sed Fluid systems.

#### **3.5.4 Applications of CMC:**

CMC has applications in various industries such as

- Textile Industry
- Paint Industry
- Soaps & Detergents
- Pesticides
- Oil Well Drilling
- Paper &
- Corrugated Board Industry and Ceramic Industries.

## **3.6 Titanium dioxide (TiO<sub>2</sub>)**

Titanium dioxide is a naturally occurring oxide of the element titanium. Also referred to as titanium (IV) oxide or titania, this substance also occurs naturally as three mineral compounds known as anatase, brookite, and rutile. However, it is most commonly extracted from titanium tetrachloride by carbon reduction and re-oxidization. Alternatively, it may be processed from another oxide called ilmenite, which is subjected to reduction with sulfuric acid to achieve pure titanium dioxide [56].



Figure 3.8: Titanium dioxide (Tio2)

## **3.6.1 Structure of TiO<sub>2</sub>:**

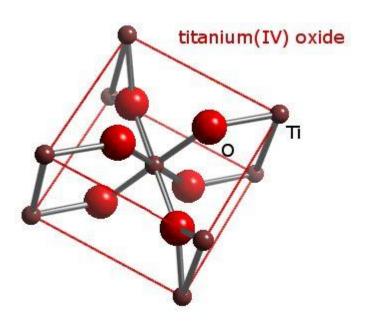


Figure 3.9: Crystal structure of Titanium (IV) dioxide (Tio2)

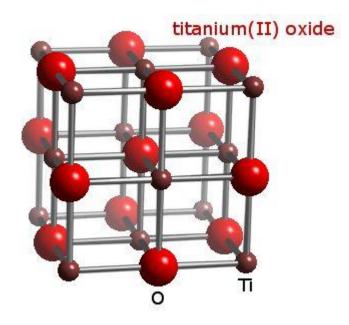


Figure 3.10: Crystal structure of Titanium (II) dioxide (Tio2)

#### **3.6.2 Production of TiO**<sub>2</sub>

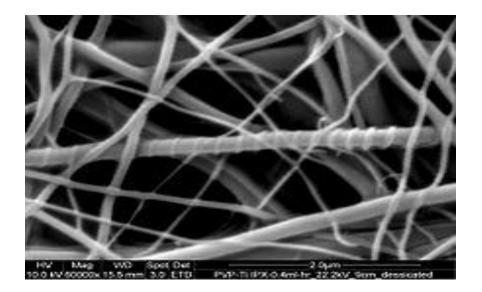
Crude titanium dioxide is purified via converting to titanium tetrachloride in the chloride process. In this process, the crude ore (containing at least 70%  $TiO_2$ ) is reduced with carbon, oxidized with chlorine to give titanium tetrachloride; i.e., carbothermal chlorination. This titanium tetrachloride is distilled, and re-oxidized in a pure oxygen flame or plasma at 1500–2000 K to give pure titanium dioxide while also regenerating chlorine [57]. Aluminum chloride is often added to the process as a rutile promoter; the product is mostly anatase in its absence.

#### Sunscreen and UV absorber

Titanium dioxide is found in almost every sunscreen with a physical blocker because of its high refractive index, its strong UV light absorbing capabilities and its resistance to discoloration under ultraviolet light. This advantage enhances its stability and ability to protect the skin from ultraviolet light. Sunscreens designed for infants or people with sensitive skin are often based on titanium dioxide and/or zinc oxide, as these mineral UV blockers are believed to cause less skin irritation than other UV absorbing chemicals. The titanium dioxide particles used in sunscreens have to be coated with silica or alumina, because titanium dioxide creates radicals in the photo catalytic reaction. These radicals are carcinogenic, and could damage the skin.

#### **Photo catalyst**

Titanium dioxide, particularly in the anatase form, is a photo catalyst under ultraviolet (UV) light. Recently it has been found that titanium dioxide, when spiked with nitrogen ions or doped with metal oxide like tungsten trioxide, is also a photo catalyst under either visible or UV light [58]The strong oxidative potential of the positive holes oxidizes water to create hydroxyl radicals. It can also oxidize oxygen or organic materials directly. Titanium dioxide is thus added to paints, cements, windows, tiles, or other products for its sterilizing, deodorizing and anti-fouling properties and is used as a hydrolysis catalyst. It is also used in dye-sensitized solar cells, which are a type of chemical solar cell (also known as a Graetzel cell).



**0Fig-3.11:** TiO<sub>2</sub> fibers and spirals

Titanium dioxide has potential for use in energy production: as a photo catalyst, it can

• Carry out hydrolysis; i.e., break water into hydrogen and oxygen. Were the hydrogen collected, it could be used as a fuel. The efficiency of this process can be greatly improved by doping the oxide with carbon. Further efficiency and durability has been

obtained by introducing disorder to the lattice structure of the surface layer of titanium dioxide Nano crystals, permitting infrared absorption.

 Titanium dioxide can also produce electricity when in nanoparticle form. Research suggests that by using these nanoparticles to form the pixels of a screen, they generate electricity when transparent and under the influence of light. If subjected to electricity on the other hand, the nanoparticles blacken, forming the basic characteristics of a LCD screen. According to creator Zoran Radivojevic, Nokia has already built a functional 200-by-200-pixel monochromatic screen which is energetically selfsufficient.

 $TiO_2$  offers great potential as an industrial technology for detoxification or remediation of wastewater due to several factors.

- 1. The process occurs under ambient conditions very slowly; direct UV light exposure increases the rate of reaction.
- 2. The formation of photo cyclized intermediate products, unlike direct photolysis techniques, is avoided.
- 3. Oxidation of the substrates to  $CO_2$  is complete.
- 4. The photo catalyst is inexpensive and has a high turnover.
- 5.  $TiO_2$  can be supported on suitable reactor substrates.

#### Pigment

Titanium dioxide is the most widely used white pigment because of its brightness and very high refractive index (n = 2.7), in which it is surpassed only by a few other materials. Approximately 4 million tons of pigmentary TiO<sub>2</sub> are consumed annually worldwide. When deposited as a thin film, its refractive index and colour make it an excellent reflective optical coating for dielectric mirrors and some gemstones like "mystic fire topaz". TiO<sub>2</sub> is also an effective opacifier in powder form, where it is employed as a pigment to provide whiteness and opacity to products such as paints, coatings, plastics, papers, inks, foods, medicines (i.e. pills and tablets) as well as most toothpaste. In paint, it is often referred to offhandedly as "the perfect white", "the whitest white", or other similar terms. Opacity is improved by optimal sizing of the titanium dioxide particles.

Titanium dioxide is often used to whiten skimmed milk; this has been shown statistically to increase skimmed milk's palatability [59].

# 3.7 Natural dye

**Natural dyes** are dyes or colorants derived from plants, invertebrates, or minerals. The majority of natural dyes are vegetable dyes from plant sources – roots, berries, bark, leaves, and wood — and other organic sources such as fungi and lichens.

Archaeologists have found evidence of textile dyeing dating back to the Neolithic period. In China, dyeing with plants, barks and insects has been traced back more than 5,000 years [60]. The essential process of dyeing changed little over time. Typically, the dye material is put in a pot of water and then the textiles to be dyed are added to the pot, which is heated and stirred until the color is transferred. Textile fiber may be dyed before spinning (*dyed in the wool*), but most textiles are *yarn-dyed* or *piece-dyed* after weaving. Many natural dyes require the use of chemicals called mordant to bind the dye to the textile fibers; tannin from oak galls, salt, natural alum, vinegar, and ammonia from stale urine were used by early dyers. Many mordant, and some dyes themselves, produce strong odors, and large-scale dye works were often isolated in their own districts [61].



Figure 3.12: Red amaranth dye



Figure 3.13: Red amaranth is used as a source of deep red dye

The discovery of man-made synthetic dyes in the mid-19th century triggered the end of the large-scale market for natural dyes. Synthetic dyes, which could be produced in large quantities, quickly superseded natural dyes for the commercial textile production enabled by the industrial revolution, and unlike natural dyes, were suitable for the synthetic fibers that followed. Artists of the *Arts and Crafts Movement* preferred the pure shades and subtle variability of natural dyes, which mellow with age but preserve their true colors, unlike early synthetic dyes and helped ensure that the old European techniques for dyeing and printing with natural dyestuffs were preserved for use by home and craft dyers. Natural dyeing techniques are also preserved by artisans in traditional cultures around the world.

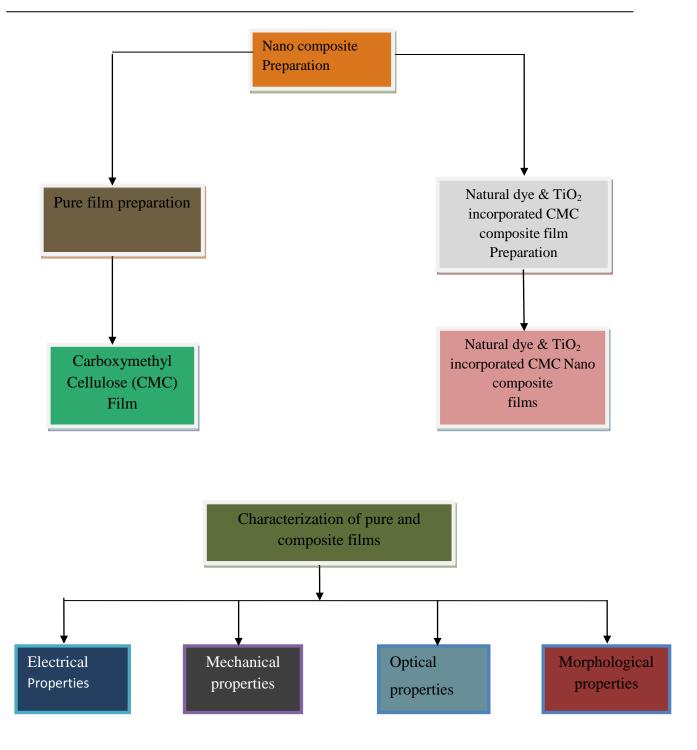
# CHAPTER FOUR EXPERIMENTAL PROCEDURES

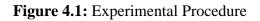
# 4.1 Introduction:

The chapter deals with the materials that we have used in this experiment, the instruments name & its model number and the whole procedures. The preparation and characterization of Titanium dioxide (TiO<sub>2</sub>) and natural dye incorporated with the polymer Nano composites have been discussed in this chapter also. The experimental setup of this research work and the preparation to make the mixture with dye and without dye are also given in here. The mixture is converted then into the film by solution casting. The best film changing with some parameters has been shown in here. Then, all the films before mixing the dye and after mixing the dye were characterized by Electrical properties (IV curve, Conductivity vs. Resistance, Voltage vs. Resistance), Mechanical properties (Tensile strength, Elongation break, E-Modulus, Hardness), Optical properties (UV test) and Morphological properties. Each of the method has been explained with their own principles.

<b>Yable 4.1:</b> Methods of characterization of the Nano composites films
--

Characterization Parameters	Methods
Electrical properties	Current(I), Voltage(V), Conductivity(G),
	Resistance(R), Inductance(L)
Mechanical properties	Tensile strength(TS), Elongation break(Eb),
	E-Modulus(E-m), Hardness(H)
Optical properties	UV test
Morphological properties	Microscopic test





#### **4.2 Materials**

In this research project, Carboxymethyl cellulose (CMC), Titanium dioxide (TiO<sub>2</sub>) and natural dye were used. CMC was purchased from Hatkhola, Bangladesh and TiO<sub>2</sub> was purchased from industrial suppliers. The molecular weight of TiO<sub>2</sub> used in this work is 79.866 g/mol.

#### 4.3 Methods

# 4.3.1 Preparation of pure carboxymethyl cellulose (CMC), Titanium dioxide ( $TiO_2$ ) incorporated with the carboxymethyl cellulose (CMC) Nano composites

All the membranes or films of pure carboxymethyl cellulose (CMC) and Titanium dioxide (TiO<sub>2</sub>) incorporated with the CMC blends were prepared by using casting solution technique. To prepare pure CMC film, we had dissolved 2% CMC in 200mL distill water. Then it was magnetically stirred for 30 minutes at 45°C in the stirring machine until CMC is dissolved in the distill water (**figure4.2 & 4.3**). Then four 50mL beakers were taken. The total 200mL dissolved solution was divided into the four beakers equally. So, each of the four beakers carried out 50mL dissolved solution (**figure 4.4**). After that TiO<sub>2</sub> was added in the 0.02gm, 0.05gm, 0.1gm, 0.2gm ratio in the dissolved Nano composite films (**figure 4.5**). But in the pure solution, there were no mixture of TiO<sub>2</sub>. Then, all the four beakers were magnetically stirred for 20 minutes at 30°C in the stirring machine until TiO<sub>2</sub> was dissolved in the distill water (**figure 4.6 & 4.7**). The pure aqueous solution of CMC was cast on a glass plate covered by a silicon paper (**figure 4.8 & 4.9**) and the water is allowed to evaporate at room temperature in laminar air flow for 1 day.

After 1 day, the solution kept on the glass plate covered by a silicon layer plate formed Nano composite films. Five Nano composite films were got. All the Nano composite films were good. There was no crack in the Nano composite film. All dried films were coded as I (0.2 TiO<sub>2</sub>), II (0.1 TiO<sub>2</sub>), III (0.01 TiO<sub>2</sub>), IV (0.02 TiO<sub>2</sub>), and V (pure). The film thickness was about 0.1-0.15mm.

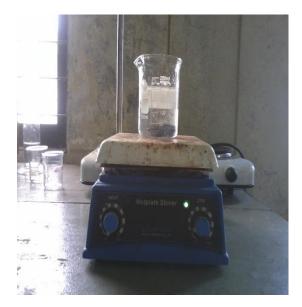


Figure 4.2: CMC was put into the beaker & Stirred



Figure 4.3: Upper view of the CMC & water

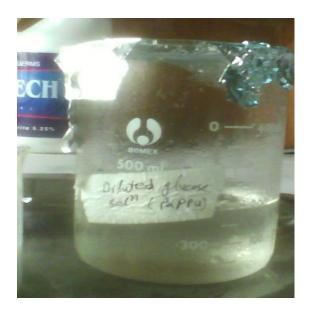


Figure 4.4: Soltution of CMC after stirring



Figure 4.5: Solution after mixing  $TiO_2$ 



Figure 4.6: TiO<sub>2</sub> mixed solution was stirred magnetically



Figure 4.7: All samples were ready to put into the layer plate



**Figure 4.8:** Solution were being poured in the poured in the layer plate



Figure 4.9: All the solution were layer plate without any dye

#### **4.3.2** Change of parameters

After getting all (five) the polymer Nano composite films, all the electrical properties had been measured of each sample. The best result was found for the Nano composite film of 0.2gm TiO<sub>2</sub>. Because the Nano composite film was fine and there was no crack (**figure 4.9**). And the current and voltage change was proportional to each other whereas the change of electrical properties in other sample was not proportional to each other.

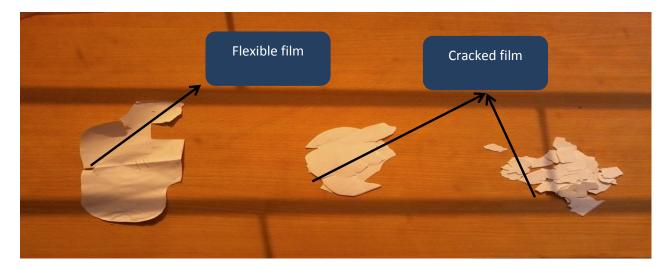


Figure 4.10: The flexible sample & cracked sample

# 4.3.3 Preparation of pure carboxymethyl cellulose (CMC), Titanium dioxide $(TiO_2)$ incorporated with the carboxymethyl cellulose (CMC) Nano composites for the Second Time

The best Nano composite film was for  $0.2 \text{gm TiO}_2$ . The change in electrical properties below the  $0.2 \text{gm TiO}_2$  was not good like this sample. Because the current-voltage changes below  $0.2 \text{gm TiO}_2$  was not proportional to each other. So, the amount of TiO<sub>2</sub> had been increased until we did not get the flexible (**figure 4.9**) and best result film than before.

# **4.3.4** Again Film Preparation to Determine the Flexible Condition of the Nano composite Film

Again, 2% CMC were dissolved in 200mL distill water to prepare pure CMC film. Then it was magnetically stirred for 30 minutes without any heating in the stirring machine until CMC is dissolved in the distill water (**figure 4.2**). This time, heat was not given in the beaker. Because there were some bubbles in the beaker because of heating. Then five 50mL beakers were taken. The total 200mL dissolved solution was divided into the five beakers equally. So, each of the five beakers carried out 50mL dissolved solution. After that  $TiO_2$  was added in the 1.00gm, 3.00gm, 5.00gm, 7.00gm & 10gm ratio in the dissolved Nano composite films (**figure 4.7**). There was no pure solution in this time. The pure aqueous solution of CMC was cast on a glass plate covered by a silicon paper and the water is allowed to evaporate at room temperature in laminar air flow for 1 day (**figure 4.8 & 4.9**).

After 1 day, the solution kept on the glass plate covered by a silicon layer plate formed Nano composite films. Five Nano composite films were got. But this time, there were some variation of among all the Nano composite films. All dried films were coded as I (1.00gm TiO<sub>2</sub>), II (3.00gm TiO<sub>2</sub>), III (5.00gm TiO<sub>2</sub>), IV (7.00gm TiO<sub>2</sub>), and V (10.00gm TiO<sub>2</sub>). The film thickness was about 0.1-0.15mm.

There were some cracks in the film of II, III, and IV & V as there was much amount of  $TiO_2$ . But the electrical properties were good of these films. As our target was to make the flexible solar cell, so the film of I was taken. And the range of using  $TiO_2$  had been known also to us.

#### 4.3.5 Film Preparation Using Natural Dye (Red Amaranth)

Again, 2% CMC were dissolved in 200mL distill water to prepare pure CMC film. Then it was magnetically stirred for 30 minutes without any heating in the stirring machine until CMC is dissolved in the distill water. Then three 50mL beakers were taken. The total 200mL dissolved solution was divided into the three beakers equally. So, each of the five beakers carried out 50mL dissolved solution. After that  $TiO_2$  was added in the 1.00gm in the dissolved Nano composite films. In this time natural dye (**figure 4.11 & 12**) was added. In the first beaker 1% dye and in the second beaker 2% dye were added. And there was no dye in the third beaker. Then the pure aqueous solution of CMC was cast on a glass plate covered

by a silicon paper and the water is allowed to evaporate at room temperature in laminar air flow for 1 day.

All the Nano composite films were good (**figure 4.14**). All dried films were coded as I (1% of natural dye), II (2% of natural dye) & III (without any dye). Then, all kinds of electrical properties, mechanical properties, optical properties & morphological properties were measured of these three Nano composite films.



Figure 4.11: Natural dye (red amaranth)



Figure 4.12: Dye was being put into the solution



Figure 4.13: Upper view of the solution



Figure 4.14: Dye was being dispersed

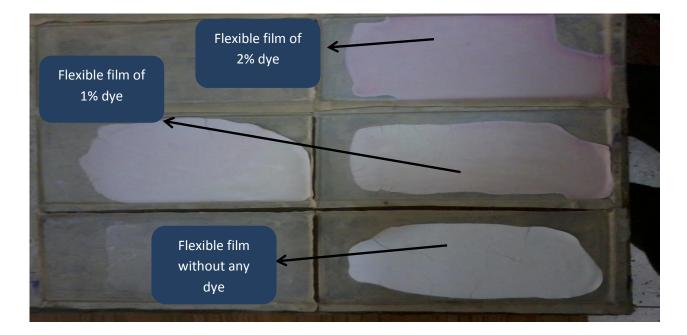


Figure 4.15: Sample were ready after drying 1 day

## 4.4 Characterization of Nano composites

#### **4.4.1 Electrical Properties**

#### **4.4.2 Instruments for Electrical Properties**

#### (a) LCR Meter

A digital LCR Meter (**figure 4.15**) was used for dc current measurement. It is a very sensitive instrument and it has versatile uses. It can measure a wide range of dc voltages, currents, resistances and electronic charges. Resistance range of this model is from 10 uOhm to 100 MOhm and capacitance range is 0.00001 pF to 99999 uF.

#### Model Number: GW INSTEK LCR-817

#### (b) Digital Multi Meter

Digital multi meter were used to measure the voltage of all the Nano composite film.

#### Model Number: GW inSTEK GDM-451

#### (c) Micro Ammeter

In this experiment micro ammeter was used to measure the current in micro range of all the Nano composite film.

#### Model Number: YOKOGAWA

#### (c) D.C. Power Supply

A Trainer Board was used for electrical measurement. This machine can give the variable voltage from 0-15.6 volt.

#### Model Number: IDL-600 ANALOG LAB

#### (d) Circuit Design

Circuit design of the electrical setup was shown in the **Figure 4.17 & 4.18**.

#### 4.4.3 Sample Preparation for Electrical Properties Measurement

The samples were cut into the square shape. The shape was 1 inch X 1 inch. Two clips were used to hold the sample.

# 4.4.4 Resistance, Quality factor, Capacitance, Dissipation factor & Inductance Measurement

Then LCR Meter was used to measure the resistance, quality factor, capacitance, dissipation factor & inductance for each of the sample. LCR Meter will give the output complete digitally.



Figure 4.16: LCR meter

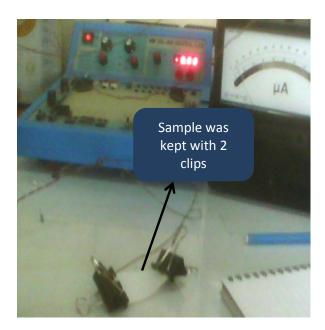


Figure 4.17: variable voltage was given in The sample & current was measured



Figure 4.18: The whole electrical equipment

#### 4.4.5 Current-Voltage Curve

Trainer board, multi meter & two clips (**figure 4.17**) were used for the measurement of current-voltage. Each of the samples was held with two clips (**figure 4.16**) which are connected with wires. The portion of these wires was connected with the micro ammeter (model number: YOKOGAWA) and variable voltage. Then, variable voltages were applied in the sample from the trainer board. After that, current and voltages were measured respectively with the help of micro ammeter and multi meter.

#### 4.4.6 Voltage vs. Resistance Measurement

The measurement of resistance of a specimen of the material is with well-defined regular square shape. The resistance was measured across each of the point of current and voltages. The following formula was used to measure the resistance of each of the sample.

#### We know, V=IR

So, R=V/I.....(4.3.1)

The Resistance was calculated from the relation 4.3.1

#### 4.4.7 Conductivity vs. Resistance Measurement

The conductivity was measured across each of the point of resistances. The following formula was used to measure the conductivity of each of the sample.

We know, RG=1

So, G=1/R......(4.3.2)

The Conductivity was calculated from the relation 4.3.2

#### **4.5 Mechanical Properties**

#### 4.5.1 Hardness Test

Rockwell Hardness Tester Model No: TQ MAT 10/RAB Load= 60kg Indenter= 1/16" Scale= Red scale Ball pointer 1/8" is unavailable, so we used 1/16"

Each of the Nano composite film of 1% dye, 2% dye and without dye incorporated with the carboxymethyl cellulose were put in the Rockwell hardness tester. Indenter 1/8'' (load 150 kg) was unavailable in the IUT mechanics lab. It is used for the soft and thin materials. In this experiment, indenter 1/16'' was used to measure the hardness of the samples (**figure 4.18**) which was available in this lab. All the Nano composite film was measured in the B scale.



Figure 4.19: Rockwell Hardness Tester

# 4.5.2 Tensile Strength, Percent Elongation at Break, E-Modulus Measurement

Instrument name: Universal Testing machine (figure 4.19) Model No: H50KS-0404, RHi-50z Setting: Load range: 500N Extension Range: 100m Gauge length: 40mm Speed: 10mm/min Each of the samples was cut into the following ratio.

Length: 6.00cm

Width: 1.00cm

The thickness of each of the sample was measured with digital slide calipers (Stainless Hardened) (**figure 4.20**). The samples were put into the universal testing machine (**figure 4.21 & 22**). After that the machine was started until the break of the sample. When, the sample was cut down, automatically the force would be zero which was shown in the monitor attached with machine. Then, tensile strength, elongation at break, e-modulus were measured of all the samples. Maximum force and break length were measured also in the universal testing machine.



Figure 4.20: Universal Testing Machine



Figure 4.21: Thickness of the sample was measured by digital slide calipers



Figure 4.22: Sample was being put in the machine



Figure 4.23: Nano composite film were kept in the universal testing machine

## **4.6 Optical Properties Measurement**

#### Instrument name: T60 UV-Visible Spectrophotometer

Ultraviolet-Visible spectroscopy (**figure 4.23**) is useful as an analytical technique for two reasons. First it can be used to identify some functional groups in molecules. Secondly, it can be used for assaying. This second role determining the content and strength of a substance is extremely useful. The gaps of optical energy, direct and indirect transitions and forbidden transitions of optically active substances can be determined from the UV-Visible spectroscopic studies for the potential applications like light guide material, optical fibers, optical coating to inhibit corrosion etc. The most important application of UV-Visible spectroscopy is to determine the presence, nature and extend of conjugation present in the material.

Each of the samples was cut into the following shape (figure 4.24).

Length: 6.00cm Width: 1.00cm

The Nano composite films were put into the tray of spectrometer (**figure 4.25**). Then, the machine was started and curve of different operating mode absorption, wavelength and emission were taken. In this way, the optical properties of Nano composite films were measured with a T-60A spectrophotometer UV-Visible spectrophotometer (PG electronics U. K.).



Figure 4.24: UV- Visible spectrophotometer

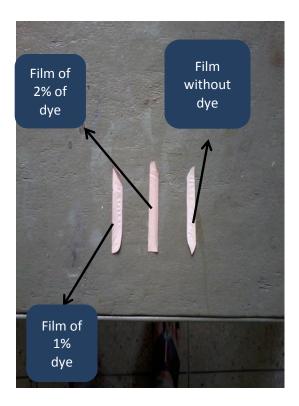


Figure 4.25: Sample were cut before putting Figure 4.26: Sample were put in the UV Into UV- Visible Spectrophotometer



-Visible Spectrophotometer

# **4.7 Morphological Properties**

### Instrument name: Inverted microscope Model No: De Winter

One portion of the Nano composite film of each sample was put into the inverted microscope (figure 4.26 & 4.27). The condition of both top side and back side view of the Nano composite film was taken with the help of inverted microscope. Well dispersion and not well dispersion were easily shown in the figure 4.28. The condition of the natural dye was also seen in the figure 4.24.



Figure 4.27: Inverted Microscope

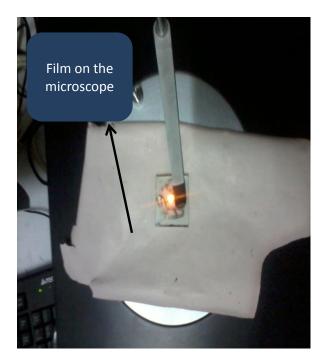


Figure 4.28: Sample was being put into the Inverted Microscope



Figure 4.29: Morphological view of the film were shown in the computer

# **CHAPTER FIVE**

# **RESULTS AND DISCUSSION**

# **5.1 Introduction**

In this chapter, the result of Nano composite film with dye and without dye incorporated with carboxymethyl cellulose (CMC) Nano composite are prepared, characterized and discussed elaborately. The electrical properties such as voltage, current, resistance, inductance, capacitance and conductivity of the Nano composite film are shown in this chapter. The UV-Visible light absorption by the sample were measured in order to investigate the optical properties. The particle morphology, view of top and rear side was studied by inverted microscope. Various physico-mechanical properties of the prepared Nano composites were measured in terms of tensile strength, elongation at break, e-modulus, maximum force, break length. Most of the data presented for physico-mechanical properties of the Nano composite films of dye related in this chapter are average values of at least three samples. Some times for the confirmation of the result the same work had done more than once and twice. In this chapter all of these investigations are discussed.

## **5.2 Electrical Properties**

#### 5.2.1 Current-Voltage (I-V) Characteristics

The I-V characteristics of  $TiO_2$  incorporated with carboxymethyl cellulose (CMC) Nano composites showed Ohmic behavior. Changing of current is proportional to the voltage changes. Besides, this can be observed from the **figure 5.8**, **5.9** & **5.10** that the current conduction increased rapidly for the sample containing higher percentage of  $TiO_2$ . It increases the current with the voltages changes, but these samples are more cracked due to increase titanium dioxide ( $TiO_2$ ). The result for using 1.00gm titanium dioxide ( $TiO_2$ ) (**figure 5.6**) gives the best output and it also works as a flexible materials. So I-V characteristics of 1.00gm  $TiO_2$  containing Nano composite film are well fitted to ohms law. The current-voltage were fluctuating which were shown in the **figure 5.1& 5.2**. But in the other case,

current-voltage was fluctuating a little bit and proportional to each other. These changes were shown in the **figure 5.3**, **5.4**, **5.5**, **5.6**, **5.7**, **5.8**, **5.9** & **5.10** 

Current-voltage changes are fluctuating a little bit. When voltages increased, current was increasing none uniformly which is shown in the **figure 5.1 & 5.2**.

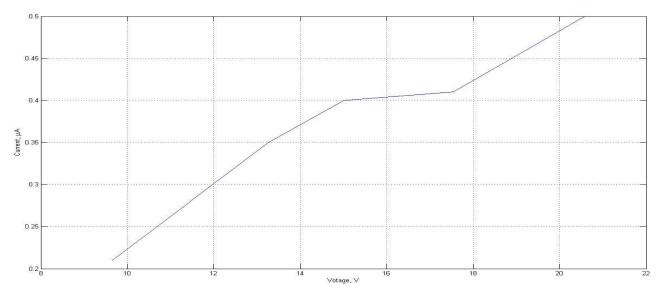
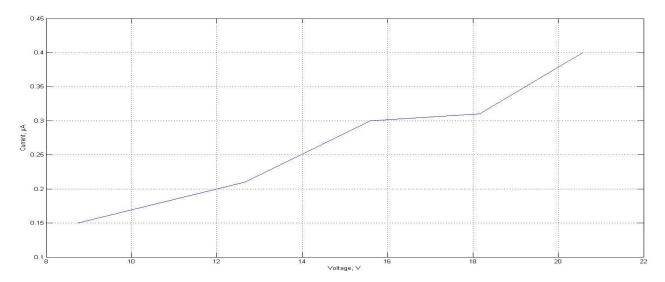
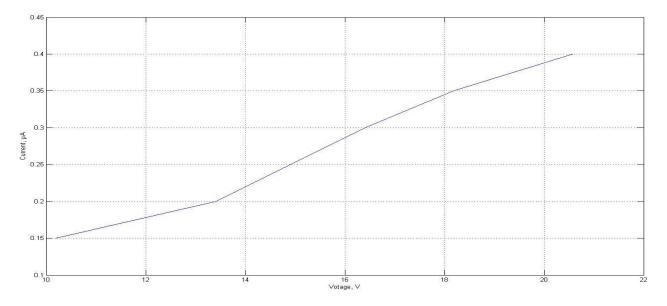


Figure 5.1: Effect of voltage on current of 0.01gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite



**Figure 5.2:** Effect of voltage on current of 0.05gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Here, Current-voltage changes are proportional to each other. When voltages increased, current was increasing uniformly with the voltages which are shown in the **figure 5.3 & 5.4**. It proves the conductivity of carboxymethyl cellulose (CMC).



**Figure 5.3:** Effect of voltage on current of 0.1gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

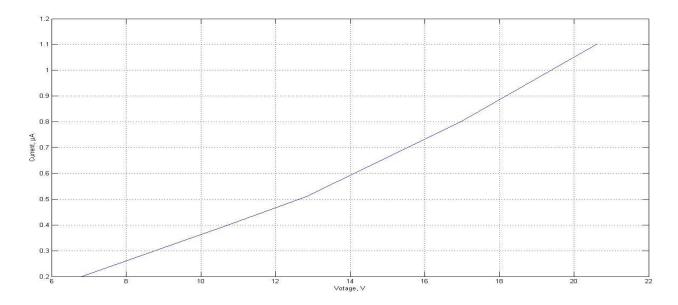
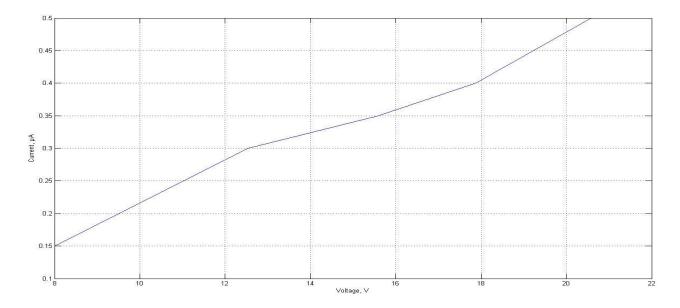


Figure 5.4: Effect of voltage on current of 0.2gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Here, Current-voltage changes are also proportional to each other. When voltages increased, current was increasing uniformly with the voltages which are shown in the **figure 5.5 & 5.6**. Besides 1gm TiO<sub>2</sub> has given the best result and flexibility of the Nano composite film. And it proves the conductivity of carboxymethyl cellulose (CMC).



**Figure 5.5:** Effect of voltage on current of pure solution without TiO<sub>2</sub> & any dye incorporated carboxymethyl cellulose Nano composite

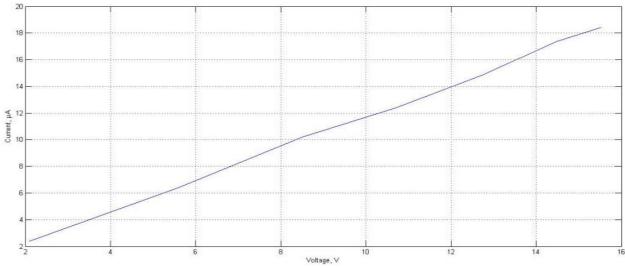
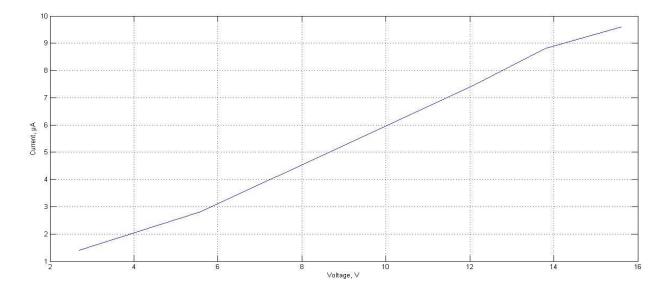


Figure 5.6: Effect of voltage on current of 1.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Here, Current-voltage changes are also proportional to each other. When voltages increased, current was increasing uniformly with the voltages which are shown in the **figure 5.7 & 5.8**. And it proves the conductivity of carboxymethyl cellulose (CMC) and titanium dioxide  $(TiO_2)$ .



**Figure 5.7:** Effect of voltage on current of 3.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

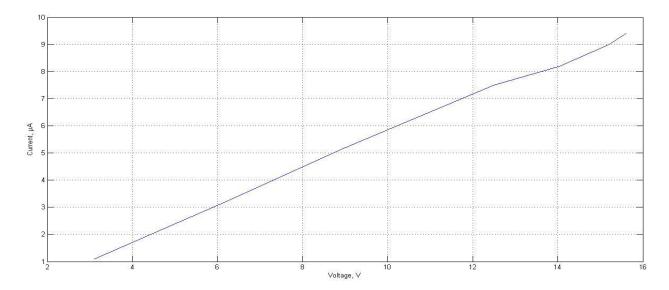
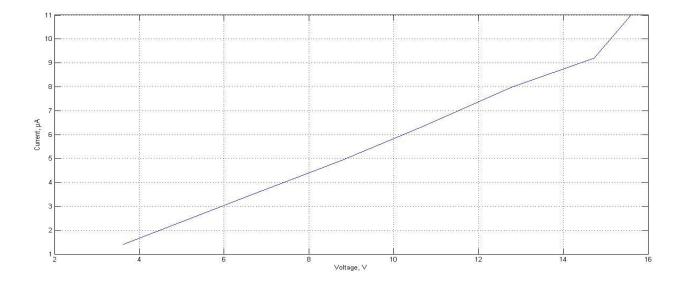


Figure 5.8: Effect of voltage on current of 5.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Here, Current-voltage changes are also proportional to each other. When voltages increased, current was increasing uniformly and rapidly with the voltages which are shown in the **figure 5.9 & 5.10.** And it proves the conductivity of carboxymethyl cellulose (CMC) and titanium dioxide (TiO<sub>2</sub>). But, in this Nano composite film, we did not get the flexible condition of the film because of increasing titanium dioxide (TiO<sub>2</sub>).



**Figure 5.9:** Effect of voltage on current of 7.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

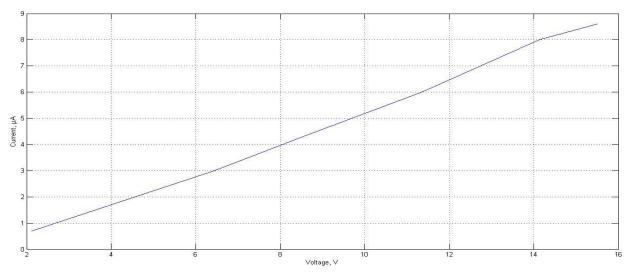


Figure 5.10: Effect of voltage on current of 10.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

# 5.2.2 Current-Voltage curve of natural dye & $TiO_2$ incorporated with carboxymethyl cellulose Nano composite

As natural 1% & 2% of natural dye were mixed in the solution incorporated with the Nano composite film. Current-voltage changes were proportional to each other of the entire sample including the pure Nano composite film without any dye. Current was increasing when voltage increasing in every case. These changes were shown in **figure 5.11, 5.12 & 5.13.** Current-voltage changing was proportional to each other for 1% of natural dye, 2% of natural dye & without dye.

Current was increasing uniformly after adding the dye in the Nano composite film when voltage was increasing too. It is shown in the **figure 11 & 12**.

Besides, current was also increasing in the Nano composite film without adding natural dye when voltage was increasing too. It is shown in the **figure 13.** 

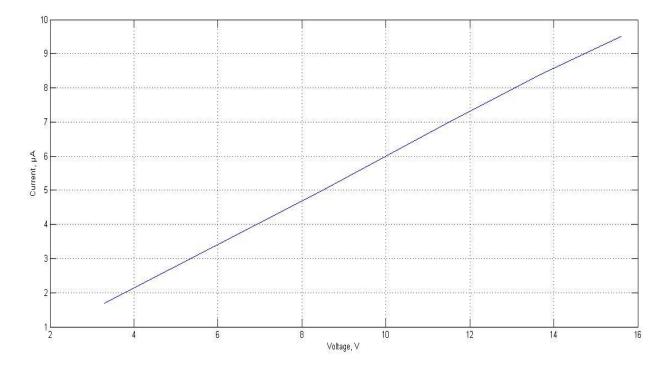
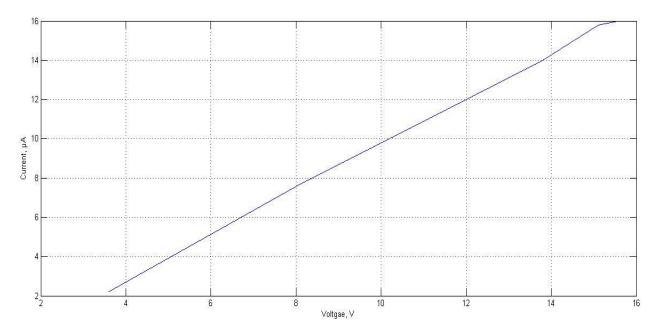
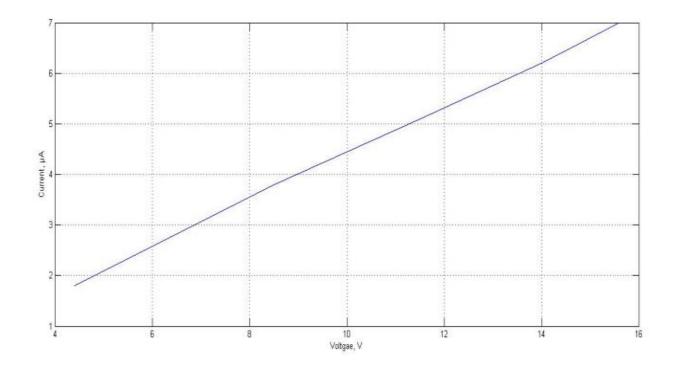


Figure 5.11: Effect of voltage on current of 1.00gm TiO<sub>2</sub> containing 1% natural dye incorporated carboxymethyl cellulose Nano composite



**Figure 5.12:** Effect of voltage on current of 1.00gm TiO<sub>2</sub> containing 2% of natural dye incorporated carboxymethyl cellulose Nano composite

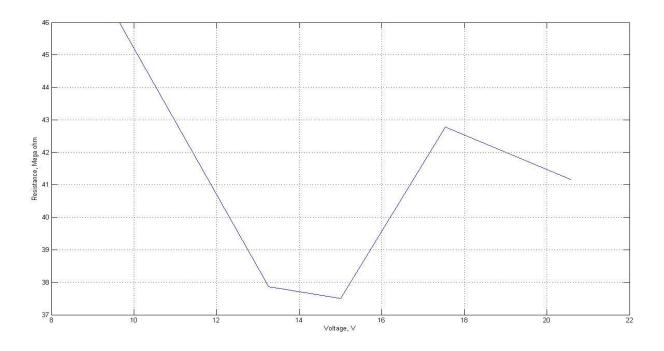


**Figure 5.13:** Effect of voltage on current of 1.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

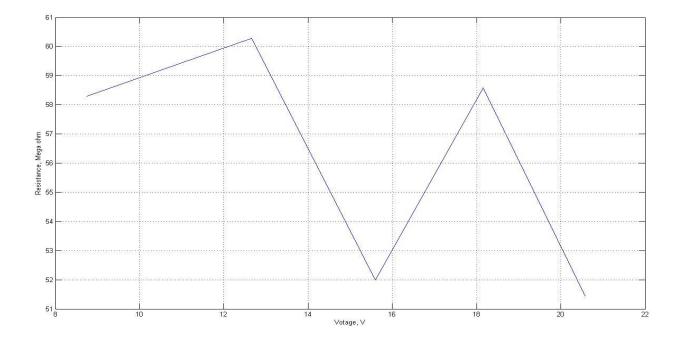
#### **5.2.3 Voltage vs. Resistance Characteristics**

Voltage and resistance changes are abnormally changed to each other for the sample of 0.01gm, 0.05 TiO<sub>2</sub> & pure solution. Here, voltage and resistance fluctuate to each other for the 0.01gm, 0.05gm TiO<sub>2</sub>. But resistance was decreasing due to increasing the voltage for the sample of 0.2gm TiO<sub>2</sub>. So, definitely it determines that this sample gives the best output. Resistance were increasing and decreasing when voltage was increasing. There were much more fluctuation between voltage & resistance which were shown in the **figure 5.14, 5.15, 5.1, 5.18, 5.19, 5.20, 5.21 & 5.23.** But in some case, resistance was decreasing when voltage increasing. These changes were shown in the **figure 5.16, 5.17 & 5.22.** And there were a little bit fluctuation between voltage-resistance.

Resistance was increasing and decreasing none uniformly when the voltage was increasing. And it is shown in the **figure 5.14 & 5.15**.

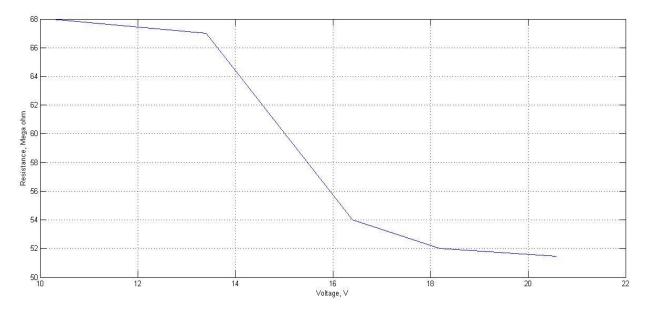


**Figure 5.14:** Effect of voltage on resistance of 0.01gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

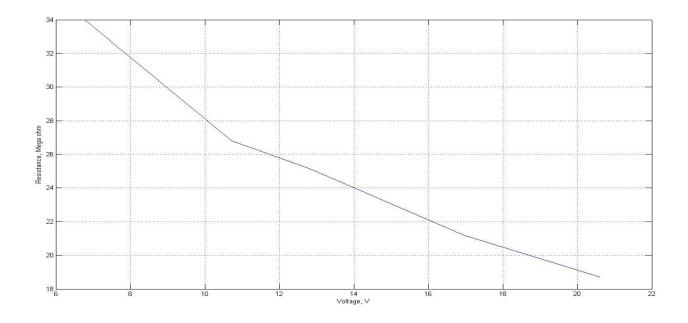


**Figure 5.15:** Effect of voltage on resistance of 0.05gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Resistance was decreasing none uniformly when the voltage was increasing (**figure 5.16**). Besides, resistance was decreasing uniformly when voltage was increasing. And it is shown in the **figure 5.17**.

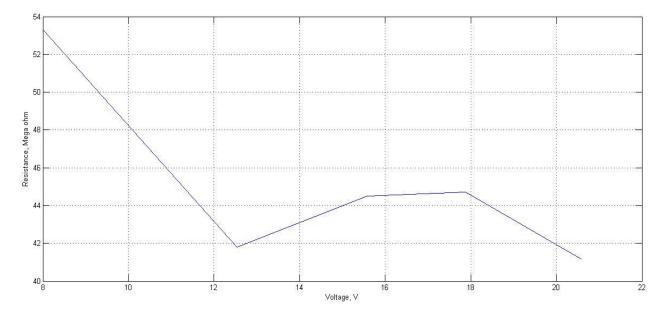


**Figure 5.16:** Effect of voltage on resistance of 0.1gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

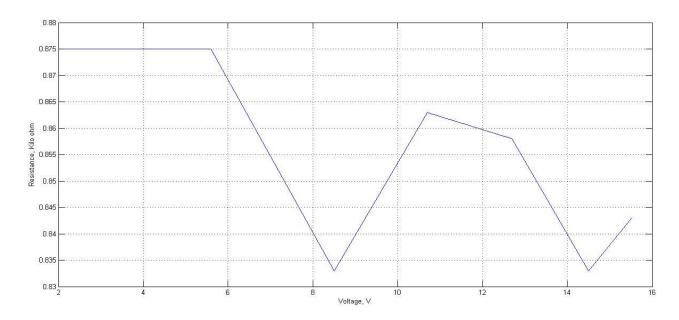


**Figure 5.17:** Effect of voltage on resistance of 0.2gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Resistance was increasing and decreasing none uniformly when the voltage was increasing. And it is shown in the **figure 5.18 & 5.19**.

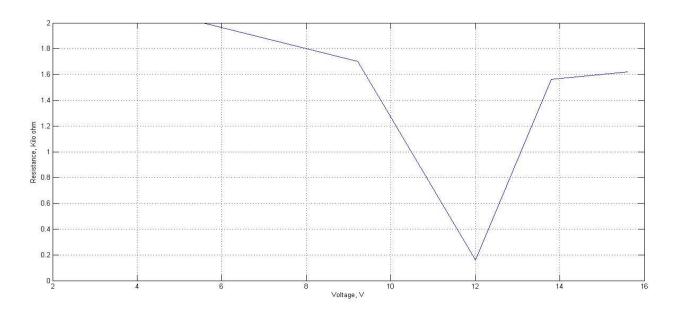


**Figure 5.18:** Effect of voltage on resistance of pure solution without any dye & TiO<sub>2</sub> incorporated carboxymethyl cellulose Nano composite

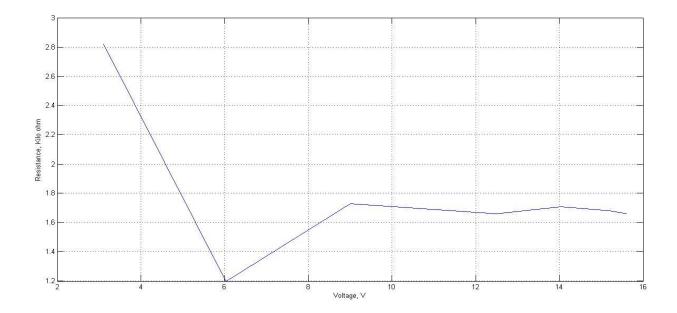


**Figure 5.19:** Effect of voltage on resistance of 1.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Resistance was increasing and decreasing none uniformly when the voltage was increasing. And it is shown in the **figure 5.20 & 5.21**.

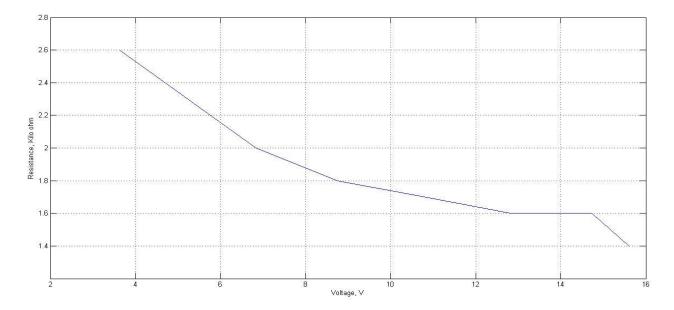


**Figure 5.20:** Effect of voltage on resistance of 3.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

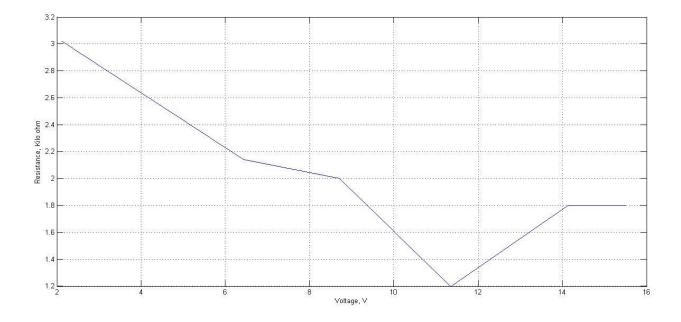


**Figure 5.21:** Effect of voltage on resistance of 5.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Resistance was decreasing uniformly (**figure 5.22**) when the voltage was increasing. Resistance was increasing and decreasing none uniformly. And it is shown in the **figure 5.23**.



**Figure 5.22:** Effect of voltage on resistance of 7.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

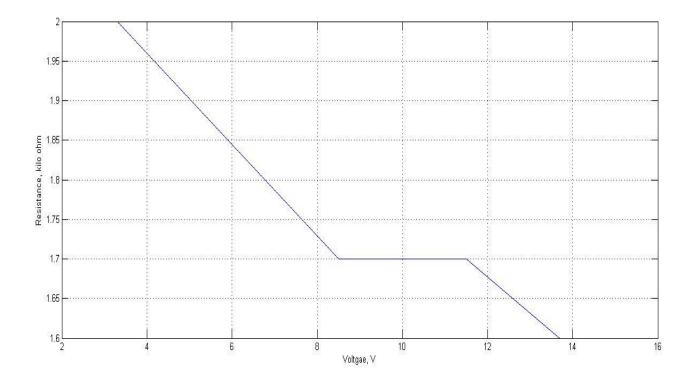


**Figure 5.23:** Effect of voltage on resistance of 10.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

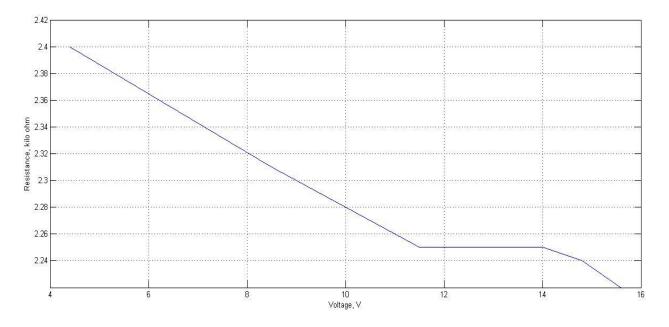
## 5.2.4 Voltage vs. Resistance Properties of natural dye & TiO<sub>2</sub> incorporated with carboxymethyl cellulose Nano composite

As like as the previous properties, voltage-resistance changes were proportional to each other of each of the sample including dye or without including dye incorporated with the carboxymethyl cellulose film. The resistance was decreasing when voltage increasing (**figure 5.24**). Resistance were constant sometime in the voltage 6-10V. But in the other case, voltage was increasing and resistance was decreasing uniformly which were shown in the **figure 5.25 & 5.26** though there was some fluctuation between voltages vs. resistance.

Resistance was fluctuating after adding 1% of natural dye in 1gm titanium dioxide and without any natural dye which is shown in **figure 5.24 & 5.26**.

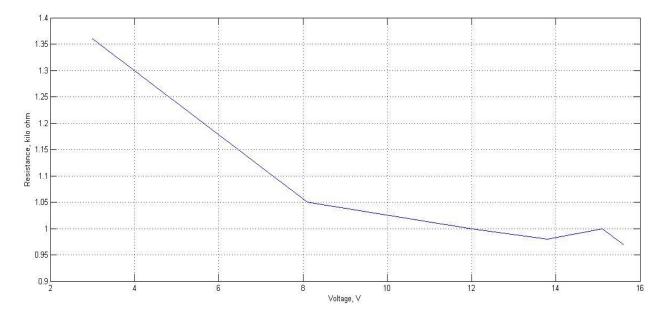


**Figure 5.24:** Effect of voltage on resistance of 1.00gm TiO<sub>2</sub> containing 1% of natural dye incorporated carboxymethyl cellulose Nano composite



**Figure 5.26:** Effect of voltage on resistance of 1.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Besides, resistance was decreasing uniformly after adding 2% of natural dye in the Nano composite film. It is shown in the **figure 5.25** 



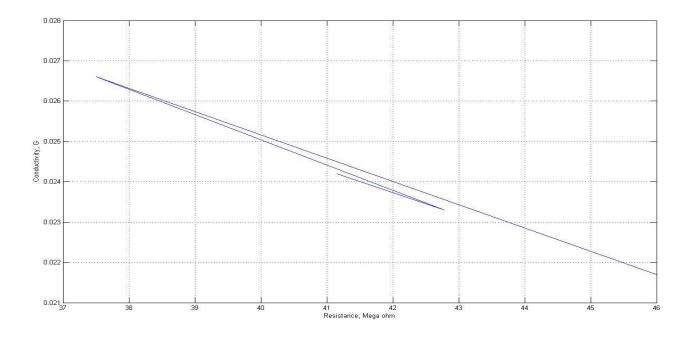
**Figure 5.25:** Effect of voltage on resistance of 1.00gm TiO<sub>2</sub> containing 2% of natural dye incorporated carboxymethyl cellulose Nano composite

#### **5.2.5 Conductivity vs. Resistance Measurement**

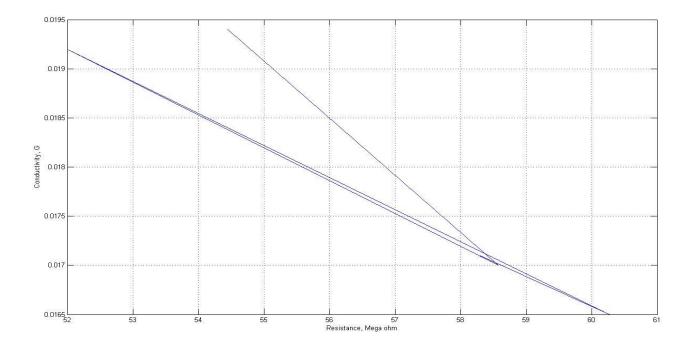
Conductivity was decreasing when resistance increased for the sample of 0.05gm TiO<sub>2</sub> & 7.00gm TiO<sub>2</sub>. But conductivity-resistance fluctuate much more for rest of the sample (0.01gm TiO<sub>2</sub>, 0.1gm TiO<sub>2</sub>, 0.2gm TiO<sub>2</sub>, 1.00gm TiO<sub>2</sub>, 3.00gm TiO<sub>2</sub>, 7.00gm TiO<sub>2</sub>, 10.00gm TiO<sub>2</sub> & the pure Nano composite film.

There were much more fluctuation between conductivity & resistance which were shown in the **figure 5.27**, **5.28**, **5.30**, **5.31**, **5.32**, **5.33**, **5.34** & **5.23**. Sometimes, conductivity was increasing or decreasing with increasing the resistance. But in some case, resistance was increasing and conductivity decreasing. These changes were shown in the **figure 5.29**, **5.35** & **5.36**. And there were a little bit fluctuation between conductivity-resistance.

Conductivity was increasing when resistance was decreasing. It was fluctuating between resistance and conductivity which is shown in the **figure 5.27 & 5.28**.

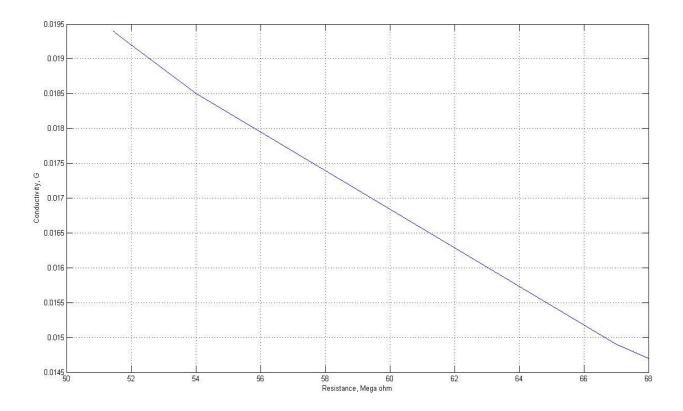


**Figure 5.27:** Effect of resistance on conductivity of 0.01gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite



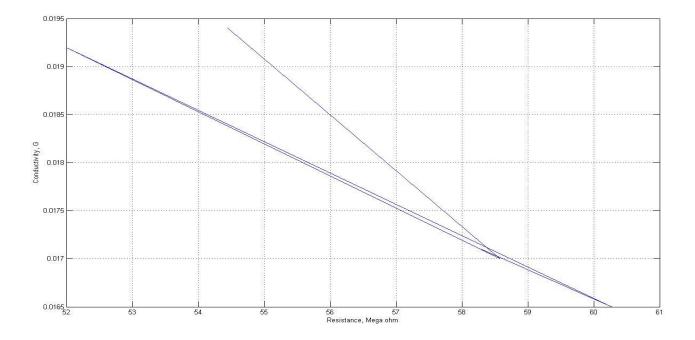
**Figure 5.28:** Effect of resistance on conductivity of 0.05gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

Resistance-conductivity changes were uniformly for adding 0.1gm titanium dioxide in the nano composite film. And it is shown in the **figure 5.29**.

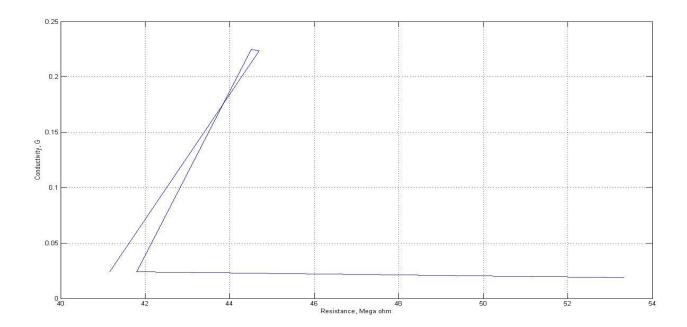


**Figure 5.29:** Effect of resistance on conductivity of 0.1gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

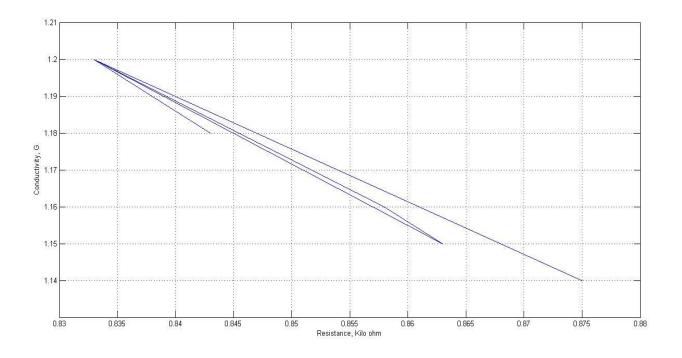
Conductivity was increasing when resistance was decreasing. It was fluctuating between resistance and conductivity which is shown in the **figure 5.30**, **5.31**, **5.32**, **5.33** & **5.34**.



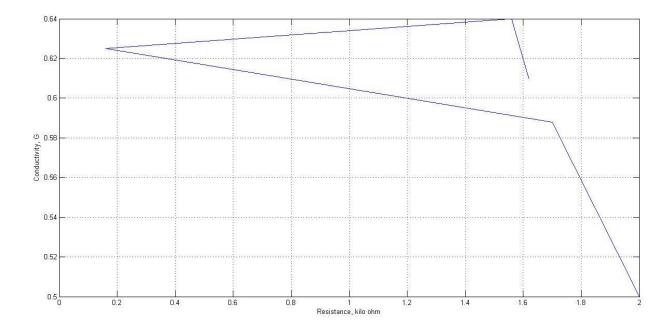
**Figure 5.30:** Effect of resistance on conductivity of 0.2gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite



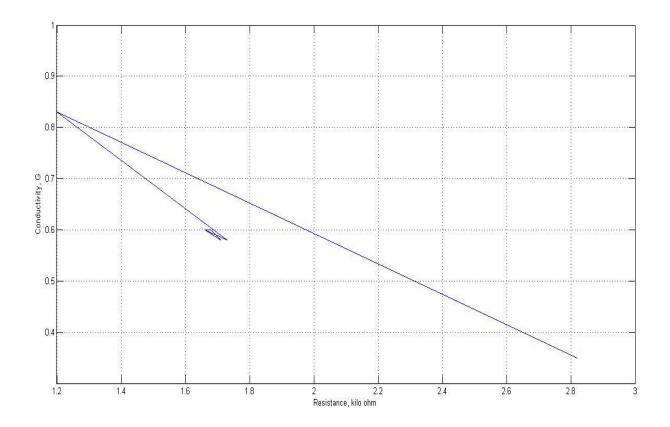
**Figure 5.31:** Effect of resistance on conductivity of TiO<sub>2</sub> without any dye & TiO<sub>2</sub> incorporated carboxymethyl cellulose Nano composite



**Figure 5.32:** Effect of resistance on conductivity of 1.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite



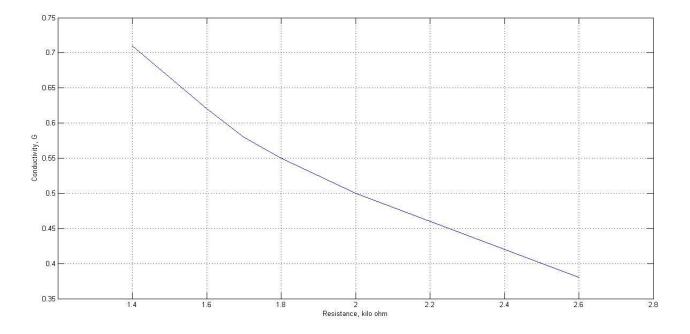
**Figure 5.33:** Effect of resistance on conductivity of 3.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite



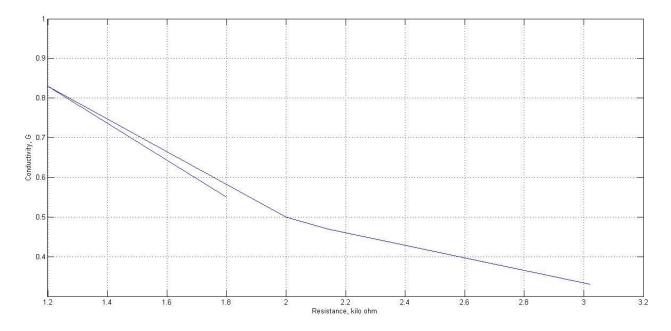
**Figure 5.34:** Effect of resistance on conductivity of 5.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

On the other hand, conductivity was decreasing uniformly when the resistances were increasing.

It is shown in the **figure 5.35 & 5.36**.



**Figure 5.35:** Effect of resistance on conductivity of 7.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite



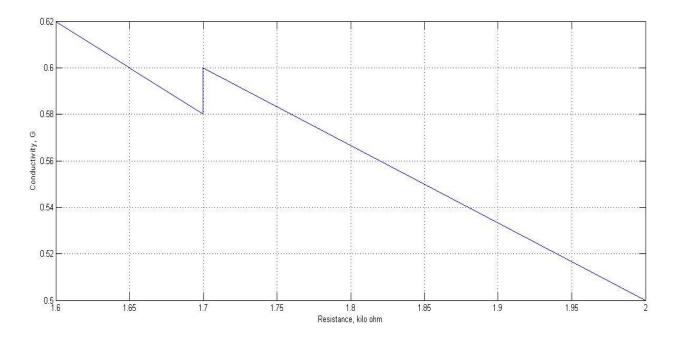
**Figure 5.36:** Effect of resistance on conductivity of 10.00gm TiO<sub>2</sub> without any dye incorporated carboxymethyl cellulose Nano composite

# 5.2.6 Conductivity-Resistance Properties of natural dye & TiO<sub>2</sub> incorporated with carboxymethyl cellulose Nano composite

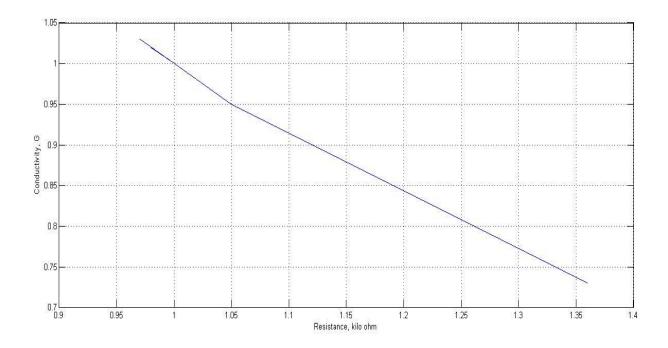
Here, the conductivity & resistance changes proportionally to each other (2% of natural dye & pure). When the resistance was increasing, conductivity was decreased also. But conductivity-resistance was fluctuating a little bit for the sample of 1% of natural dye. There were fluctuation between resistance and conductivity for the 1% of natural dye which was shown in the **figure 5.37.** In other Nano composite film, resistance was increasing and conductivity decreasing uniformly. These were shown in the **figure 5.38 & 5.39.** 

There was a little bit fluctuation after adding natural dye 1% of natural dye and without dye in the nano composite film. It is shown in the **figure 5.37 & 5.39**.

Besides, resistance-voltages changes were proportional to each other for the 2% of natural dye in the nano composite film which is shown in the **figure 5.38**.



**Figure 5.37:** Effect of resistance on conductivity of 1.00gm TiO<sub>2</sub> containing 1% of natural dye incorporated carboxymethyl cellulose Nano composite



**Figure 5.38:** Effect of resistance on conductivity of 1.00gm TiO<sub>2</sub> containing 2% of natural dye incorporated carboxymethyl cellulose Nano composite

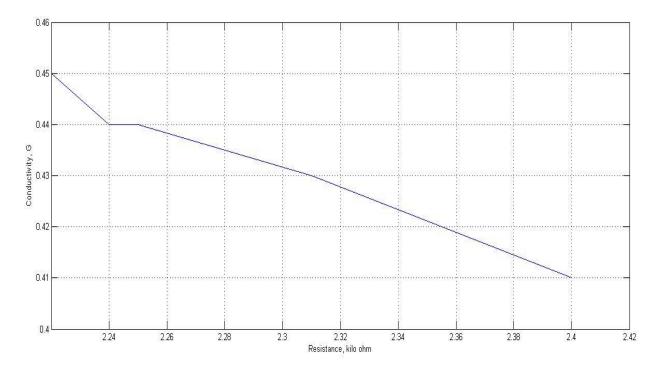


Figure 5.39: Effect of resistance on conductivity of 1.00gm TiO<sub>2</sub> without any natural dye incorporated carboxymethyl cellulose Nano composite

## **5.3 Mechanical Properties**

#### 5.3.1 Tensile Strength

Tensile strength was taken for the dye related sample (1%dye, 2%dye & pure Nano composite film).

**Figure 5.40** shows the change in tensile strength with increasing percentage of natural dye content in carboxymethyl cellulose (CMC) Nano composite films. The ultimate tensile strength of the blend films containing higher percentage of natural dye is of decreasing tendency (**figure 5.40**). After increase in tensile strength (5.00 MPa) at 1.00% of natural dye containing films, the tensile strength of the blend films began decreasing steadily with the increasing percentage of natural dye in the blend films. This must be due to repulsion between the free electron of the donor natural dye and the bonding may become weak.

After increase in tensile strength (5.00 MPa) at 100Mpa E-modulus, the tensile strength of the blend films began decreasing steadily with the increasing E-modulus. And the elongation break became constant at 5.00Mpa tensile strength.

#### **5.3.2 Elongation at Break**

The elongation at break was decreased with adding natural dye in the Nano composite film which is shown in the **Figure 5.42.** After that it was being increased till pure film and again decreased to 1.00% of natural dye containing film. The 2.00% of natural dye containing Nano composite film became brittle and showed the lowest elongation at break.

Besides, figure 5.41 shows tensile strength was decreasing when E-modulus increasing.

Figure 5.43 shows E-modulus was increasing when percentage of dye increasing.

**Figure 5.44** shows the breaking force applied on the nano composite film and its extension due to the force.

Figure 5.45 shows tensile strength was constant when elongation at break increasing.

### For 1% of dye

The Nano composite film was cut in the following ratio:

Length: 2.5inch Width: 1.0cm

Mean thickness: 0.13mm Mean width: 10.74mm Mean tensile strength: 5.0Mpa Mean elongation break: 5.16 % Mean E-modulus: 89.2 Mpa Mean Maximum Force, N: 20N Mean Break, mm: 6.21mm

#### For 2% of dye

The Nano composite film was cut in the following ratio:

Length: 2.5inch Width: 1.0cm

Mean thickness: 0.13mm Mean width: 12.00mm Mean tensile strength: 3.13Mpa Mean elongation break: 3.4 % Mean E-modulus: 218.78Mpa Mean Maximum Force, N: 5N Mean Break, mm: 1.38mm

### For pure Nano composite film

The Nano composite film was cut in the following ratio:

Length: 2.5inch Width: 1.0cm

Mean thickness: 5.1mm Mean width: 10.14mm Mean tensile strength: 4.85Mpa Mean elongation break: 19.12 % Mean E-modulus: 105.83Mpa Mean Maximum Force, N: 6.7N Mean Break, mm: 2.55mm

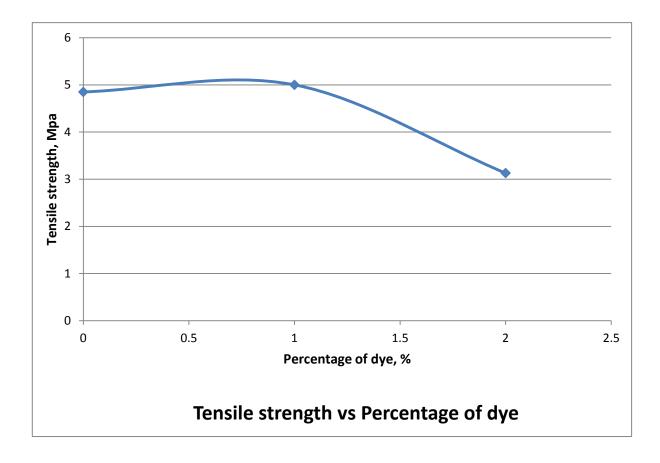


Figure 5.40: Effect of % TiO<sub>2</sub> in carboxymethyl cellulose on tensile strength

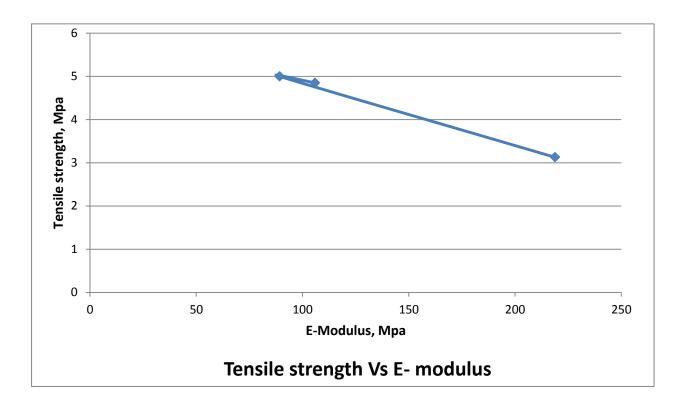


Figure 5.41: Effect of E-Modulus in carboxymethyl cellulose on tensile strength

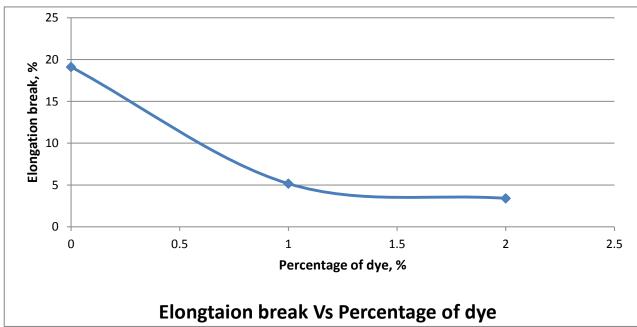
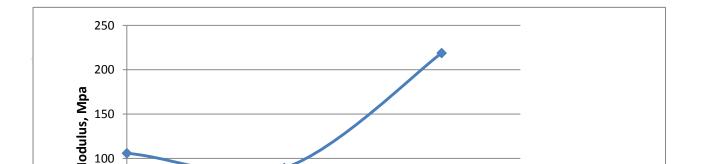
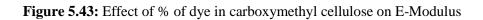


Figure 5.42: Effect of percentage of dye in carboxymethyl cellulose on elongation at break





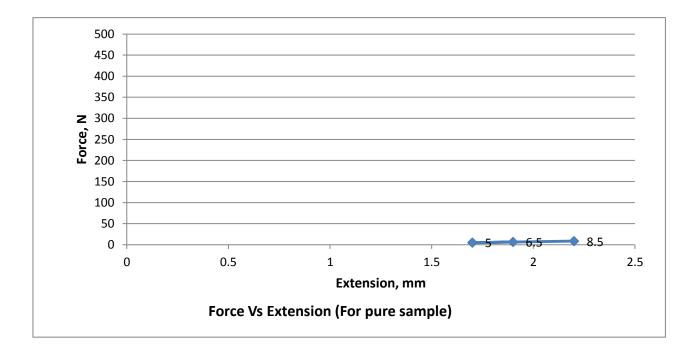


Figure 5.44: Measurement of extension of each of the film & its breaking force

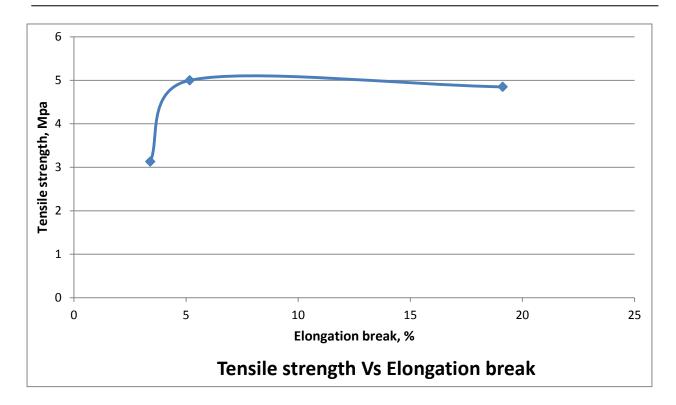


Figure 5.45: Effect of elongation at break in carboxymethyl cellulose on tensile strength

#### 5.3.3 Hardness Test

Each of the Nano composite films were taken hardness test three times. So, the value of the three times were given in the below for each of the sample. After that the mean value of each of the sample was given also.

#### For 1% of natural dye

Hardness is given below:

- B25
- B27
- B25.5

So, Mean Hardness for the 1% of natural dye is B25.8

#### For 2% of natural dye

Hardness is given below:

- B24
- **B**23
- **B**24

So, Mean Hardness for the 2% of natural dye is B23.7

#### For pure sample without any dye

Hardness is given below:

B25B25

■ B25

So, Mean Hardness for the pure Nano composite film without including any dye is B25

## **5.4 Optical Properties Analysis**

UV-Visible absorption spectra are shown in **figure. 5.46, 5.47, 5.48**. The comparisons of all the absorption were shown in the **figure 5.49**. The absorption spectrum shows a number of absorption bands corresponding to structural groups within the molecule. For pure Nano composite film the absorption remains high till 220 nm wavelength but with inclusion of TiO<sub>2</sub> & natural dye the  $\lambda_{max}$  shifts to the greater value. Absorption of TiO<sub>2</sub> containing natural dye incorporated carboxymethyl cellulose Nano composite films represents an increase degree of conjugation present in the composite. Then, all the samples were shown in one graph for the comparison. It is well known that increasing conjugation and basically it moves the absorption to longer wavelength. For pure carboxymethyl cellulose the absorption peak appear at 220 nm. The increasing absorption of the Nano composite films around wavelengths 425nm implying that absorption is basically occurred due to the  $\pi$ - $\pi^*$  absorption of polymer.

**Figure 5.46** shows some peak values of the Nano composite film for 1% of natural dye. So natural dye can absorb the light in between wavelength 300-600nm and produce energy.

**Figure 5.47** shows some peak values of the Nano composite film for 2% of natural dye. So natural dye can absorb the light in between wavelength 300-600nm and produce much energy.

**Figure 5.48** shows a lot of peak values. So, This Nano composite film without any dye can produce energy.

Figure 5.49 shows the comparison of all the Nano composite film with dye and without dye.

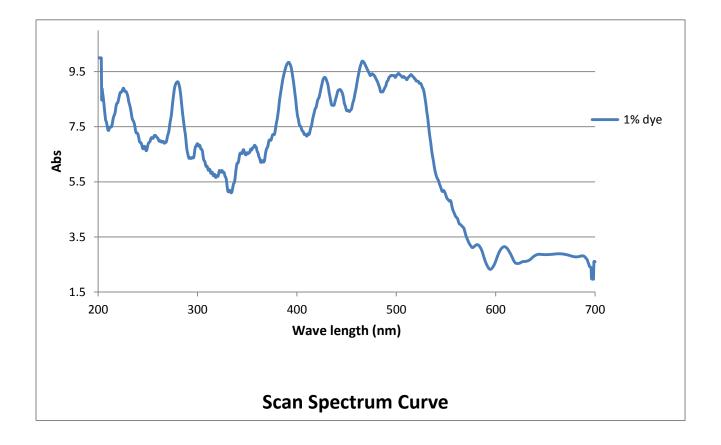
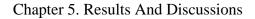
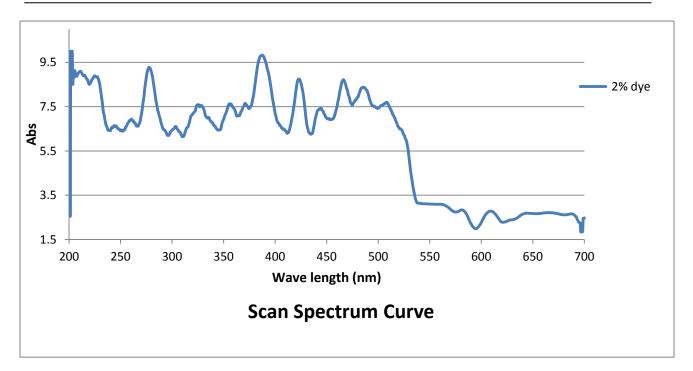
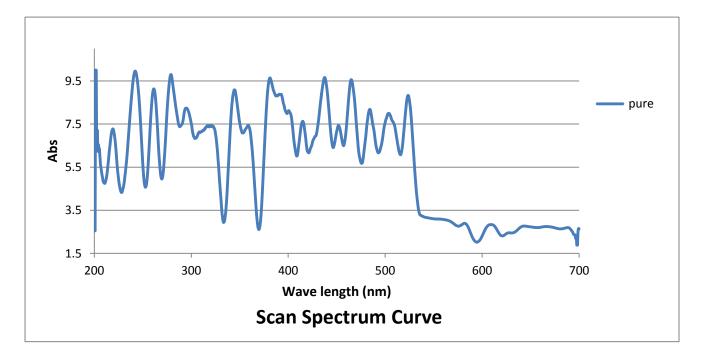


Figure 5.46: UV- Absorbance for 1.00gm TiO<sub>2</sub> & 1% of natural dye incorporated carboxymethyl cellulose film

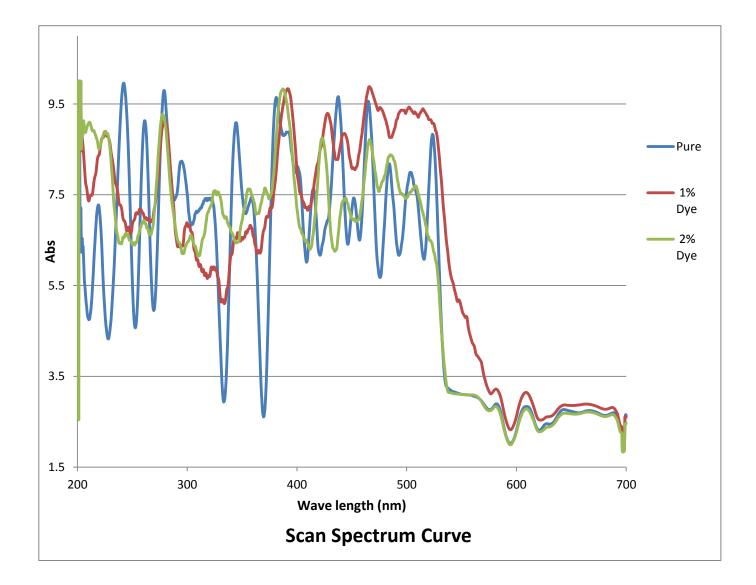




**Figure 5.47:** UV- Absorbance for 1.00gm TiO<sub>2</sub> & 2% of natural dye incorporated carboxymethyl cellulose film



**Figure 5.48:** UV- Absorbance for 1.00gm TiO<sub>2</sub> & without any dye incorporated carboxymethyl cellulose film



## Figure 5.49: Comparison of UV- Absorbance for 1%, 2% of natural dye and without dye incorporated carboxymethyl cellulose film

## **5.5 Morphological Study**

The morphologies of pure carboxymethyl cellulose,  $TiO_2$  and natural dye incorporated carboxymethyl cellulose Nano composites films are shown in the figures from **figure 5.50 to 5.54** (top side) and **figure 5.55 to 5.58** (rear side).

From inverted microscope, it is obvious that the surface is rough and uneven. The inclusion of different percentage of dye changes the roughness of the surface anymore.

## TOP SIDE

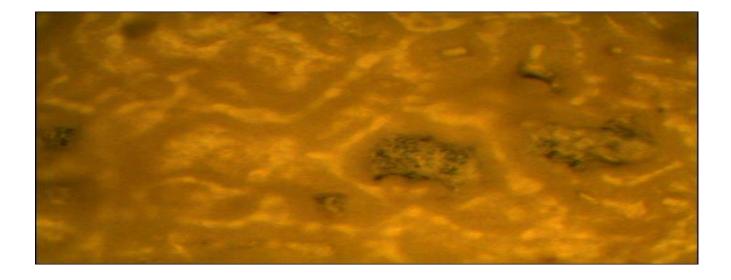
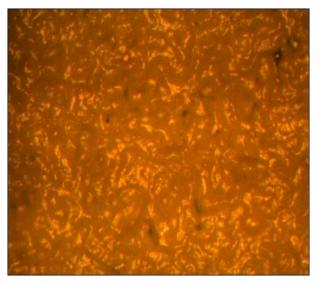


Figure 5.50: View of 1% natural dye in corporated with carboxymethyl cellulose with 1gm  $TiO_2$ 



**Figure 5.51:** 2% of naural dye (100x) with CMC & TiO<sub>2</sub>

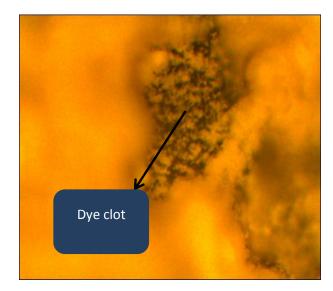
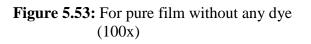
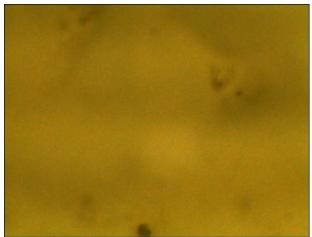


Figure 5.52: 2% of natural dye (1000x) with CMC & TiO<sub>2</sub>

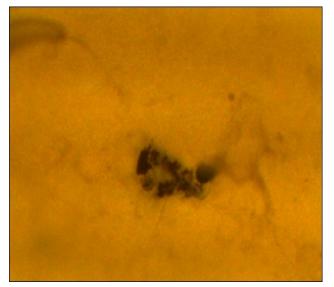




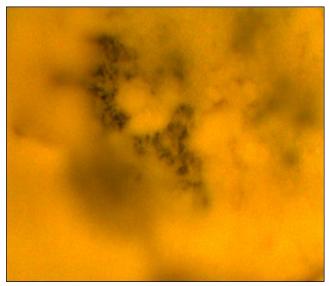


**Figure 5.54:** For pure film without any dye (1000x)

## **REAR SIDE**



**Figure 5.55:** 1% of dye (100x) with CMC & TiO<sub>2</sub>



**Figure 5.56:** 1% of dye (1000x) with CMC & & TiO<sub>2</sub>

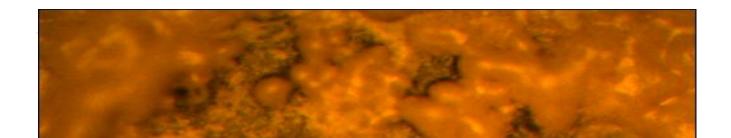


Figure 5.57: View of 2% natural dye in corporated carboxymethyl cellulose with 1gm TiO<sub>2</sub> (100x)

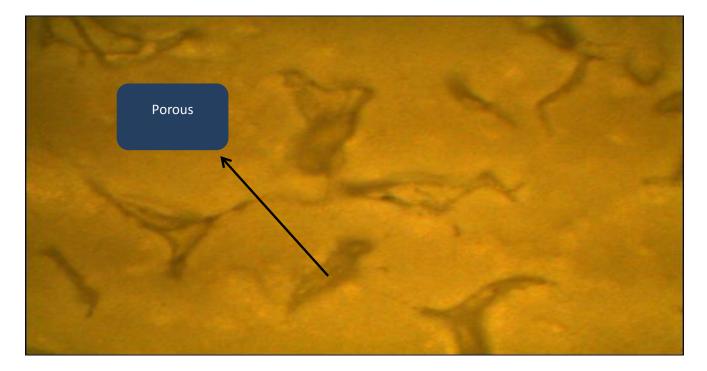


Figure 5.58: For pure film without any dye (100x) including 1gm TiO<sub>2</sub>

## CHAPTER SIX

## Conclusion

Titanium dioxide and natural dye incorporated with carboxymethyl cellulose (CMC) Nano composite give an outstanding idea to fabricate the idea of polymer based solid state dye sensitized organic solar cell. Amount of titanium dioxide determines the flexibility of the Nano composite films. Electrical properties of the Nano composite film were good for the particular film, but it is completely evident that the behavior of all Nano composite film is ohmic. It shows that it will work as a conductive material. Mechanical properties show hardness of the Nano composite film. It also shows that tensile strength of the films was decreasing with increasing the amount of natural dye. Besides E-modulus determine the stress of the Nano composite film to deform. The optical properties determine the absorption of all the three Nano composite film including natural dye and without dye. The film of 2% of natural dye shows the best result in this UV-visible test. As it can absorb the wavelengths from 300nm to 600nm in equal ratio with good peak values. Morphological test determines the dispersion of the mixture, condition of the dye clot. Both of the view of top side and rear side of the Nano composite film were taken through inverted microscope.

Throughout the research work, it has been found for 1.00gm titanium dioxide and 2% of natural dye (red amaranth) remarkably improves the electrical properties, mechanical properties and morphological properties along with increased crosslinking. Even though, this work has some outstanding results which are very important in the field of electro-active polymer, it might have great potential in the field of organic electronics especially in the field of organic semiconductor and organic photovoltaic solar cells.

## CHAPTER 7

## FUTURE WORK & FUTURE PROSPECTS

## **Future Work:**

- > To measure the different kind of test of the Nano composite film. Such as:
  - Band Gap
  - Fourier Transformed Infrared Spectroscopy (FTIR)
  - Scanning Electron Microscope (SEM)
- > To change the parameter of natural dye and carboxymethyl cellulose (CMC).
- > To fabricate electro active polymer based and solid state dye sensitized solar cell.

## **Future Prospects:**

- ➢ Thin film solar cell.
- ➢ Nano crystal or Quantum dot solar cell.
- Organic solar cell

## CHAPTER 9

## **APPENDIX**

## **Electrical Properties:**

## Table 1: 0.02gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(MQ)	Conductivity(Ω <sup>-1</sup> )
8.75	0.15	58.3	0.0171
12.66	0.21	60.28	0.0165
15.59	0.3	52	0.0192
18.16	0.31	58.58	0.0170
20.58	0.4	51.45	0.0194

## Table 2: 0.05gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(MQ)	Conductivity(Ω <sup>-1</sup> )
9.65	0.21	46	0.0217
13.26	0.35	37.86	0.0264
15	0.4	37.5	0.0266
17.54	0.41	48.78	0.0233
20.58	0.5	41.16	0.0242

### Table 3: 0.1gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(MQ)	Conductivity(Ω <sup>-1</sup> )
10.2	0.15	68	0.0147
13.4	0.2	67	0.0149
16.4	0.3	54	0.0185
18.19	0.35	52	0.0192
20.58	0.4	51.45	0.0194

### Table 4: 0.2gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(MΩ)	Conductivity(Ω <sup>-1</sup> )
6.79	0.2	33.95	0.0294
10.72	0.4	26.8	0.037
12.82	0.51	25.14	0.0397
16.96	0.8	21.2	0.047
20.6	1.1	18.72	0.0534

Voltage(V)	Current(µA)	Resistance(MΩ)	Conductivity(Ω <sup>-1</sup> )
8	0.15	53.33	0.0187
12.54	0.3	41.8	0.0239
15.58	0.35	44.51	0.2246
17.88	0.4	44.7	0.2237
20.58	0.5	41.16	0.0242

### Table 5: Pure Nano composite film

### Table 6: 1.00gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
5.6	6.4	0.875	1.14
8.5	10.2	0.833	1.2
10.7	12.4	0.863	1.15
14.5	17.4	0.833	1.2
15.52	18.4	0.843	1.18

## Table 7: 3.00gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
2.7	1.4	2	0.5
5.6	2.8	2	0.5
9.22	5.4	1.7	0.588
13.8	8.8	1.56	.64
15.6	9.6	1.62	0.61

#### Table 8: 5.00gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
6.03	3.1	1.2	0.83
9.01	5.2	1.73	0.58
12.5	7.5	1.66	0.60
14.04	8.2	1.71	0.58
15.6	9.4	1.66	0.60

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
6.83	3.6	2	0.5
8.75	4.9	1.8	0.55
10.78	6.4	1.7	0.58
12.81	8.0	1.6	0.62
15.6	11.0	1.4	0.71

### Table 9: 7.00gm TiO<sub>2</sub> with Nano composite film

### Table 10: 10.00gm TiO<sub>2</sub> with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
6.43	3.0	2.14	0.47
8.71	4.4	2.0	0.5
11.35	6.0	1.2	0.83
14.14	8.0	1.8	0.55
15.5	8.6	1.8	0.55

## Table 11: 1.00gm TiO<sub>2</sub> & 1% of natural dye incorporated with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
3.3	1.7	2.0	0.5
8.5	5.0	1.7	0.6
11.5	7.0	1.7	0.68
13.7	8.4	1.6	0.62
15.6	9.5	1.6	0.62

## Table 12: 1.00gm TiO<sub>2</sub> & 2% of natural dye incorporated with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
3	2.2	1.36	0.73
8.1	7.7	1.05	0.95
12	12.0	1.0	1.0
13.8	14.0	0.98	1.02
15.6	16.0	0.97	1.03

## Table 13: 1.00gm TiO<sub>2</sub> & without any dye incorporated with Nano composite film

Voltage(V)	Current(µA)	Resistance(kΩ)	Conductivity(Ω <sup>-1</sup> )
4.4	1.8	2.4	0.41
8.8	3.8	2.31	0.43
11.5	5.1	2.25	0.44
14	6.2	2.25	0.44
15.6	7.0	2.22	0.45

### Mechanical Properties:

### Table 14: Elongation break vs. Percentage of dye

Percentage of dye, %	Elongation break, %
0	19.12
1	5.16
2	3.4

### Table 15: E-Modulus vs. Percentage of dye

Percentage of dye, %	E-Modulus, Mpa
0	105.83
1	89.2
2	218.78

### Table 16: Force vs. Extension (For pure sample)

Force, N	Extension, mm
2.2	8.5
1.7	5
1.9	6.5

Thickness,	Width, mm	Tensile	Elongation	E-Modulus,	Maximum	Break,
mm		Strength,	Break, %	Мра	Force, N	mm
		Мра				
0.1	11.46	7.42	7.78	12.72	8.50	3.11
0.16	11.66	3.48	4.5	115.3	6.50	1.8
0.13	9.11	4.2	3.2	139.6	5.00	1.3

### Table 17: 1% of natural dye with Nano composite film

### Table 18: 2% of natural dye with Nano composite film

Thickness, mm	Width, mm	Tensile Strength, Mpa	Elongation Break, %	E-Modulus, Mpa	Maximum Force, N	Break, mm
0.12	12.00	2.4	3.6	415	3.5	1.43
0.13	12.27	3.3	3.0	107.5	5	1.2
0.15	11.62	3.7	3.6	133.8	6.5	1.5

## Table 19: Pure Nano composite film without any dye

Thickness, mm	Width, mm	Tensile Strength, Mpa	Elongation Break, %	E-Modulus, Mpa	Maximum Force, N	Break, Mm
0.17	10.66	4.69	5.70	85.8	8.50	2.28
0.15	9.62	3.47	7.20	149.5	5	2.88
0.1	10.15	6.40	6.22	82.2	6.50	2.49

### Table 20: Tensile strength vs Percentage of dye

Percentage of dye, %	Tensile strength, Mpa
0	4.85
1	5
2	3.13

### Table 21: Tensile strength Vs E- modulus

E-modulus, Mpa	Tensile strength, Mpa
105.83	4.85
89.2	5
218.78	3.13

## **Table 22: Tensile strength Vs Elongation breaks**

Elongation break, %	Tensile strength, Mpa
19.12	4.85
5.16	5
3.4	3.13

## Table 23: Some values of Nano composite film (UV Test)

Nm	Pure	1% Dye	2% Dye	nm	pure	1% dye	2% dye
700	2.633	2.602	2.474	682.8	2.645	2.781	2.62
699.6	2.658	2.611	2.443	682.4	2.642	2.779	2.617
699.2	2.631	2.61	2.448	682	2.64	2.777	2.616
698.8	2.487	2.606	2.443	681.6	2.638	2.776	2.614
698.4	1.887	1.967	1.854	681.2	2.637	2.776	2.613
698	1.884	1.966	1.848	680.8	2.637	2.776	2.613
697.6	1.882	1.969	1.837	680.4	2.637	2.776	2.614
697.2	2.184	1.984	1.83	680	2.638	2.778	2.614
696.8	2.189	1.988	1.839	679.6	2.639	2.779	2.616
696.4	2.192	1.979	1.846	679.2	2.642	2.781	2.617
696	2.355	2.39	2.246	678.8	2.644	2.783	2.62
695.6	2.359	2.398	2.253	678.4	2.647	2.786	2.622
695.2	2.362	2.407	2.263	678	2.651	2.789	2.625
694.8	2.366	2.418	2.274	677.6	2.655	2.792	2.628
694.4	2.377	2.43	2.286	677.2	2.659	2.796	2.632
694	2.416	2.471	2.326	676.8	2.664	2.8	2.636
693.6	2.456	2.513	2.366	676.4	2.669	2.804	2.64
693.2	2.496	2.555	2.406	676	2.673	2.808	2.644

692.8	2.521	2.595	2.446	675.6	2.678	2.813	2.648	
692.4	2.546	2.636	2.486	675.2	2.683	2.817	2.653	
692	2.571	2.676	2.526	674.8	2.688	2.822	2.657	
691.6	2.595	2.696	2.544	674.4	2.693	2.826	2.661	
691.2	2.618	2.714	2.562	674	2.698	2.831	2.665	
690.8	2.639	2.732	2.579	673.6	2.703	2.835	2.669	
690.4	2.656	2.749	2.595	673.2	2.707	2.839	2.673	
690	2.671	2.766	2.611	672.8	2.711	2.843	2.677	
689.6	2.682	2.78	2.624	672.4	2.715	2.846	2.68	
689.2	2.691	2.791	2.635	672	2.719	2.85	2.684	
688.8	2.696	2.8	2.644	671.6	2.722	2.853	2.687	
688.4	2.699	2.807	2.651	671.2	2.725	2.856	2.69	
688	2.699	2.812	2.655	670.8	2.728	2.859	2.693	
687.6	2.695	2.815	2.658	670.4	2.731	2.862	2.695	
687.2	2.691	2.816	2.658	670	2.733	2.865	2.698	
686.8	2.687	2.815	2.656	669.6	2.736	2.868	2.7	
686.4	2.683	2.811	2.652	669.2	2.738	2.87	2.702	
686	2.679	2.808	2.648	668.8	2.74	2.873	2.704	
685.6	2.674	2.804	2.644	668.4	2.741	2.875	2.706	
685.2	2.67	2.8	2.64	668	2.743	2.877	2.708	
684.8	2.665	2.796	2.636	667.6	2.744	2.88	2.709	
684.4	2.661	2.793	2.632	667.2	2.745	2.882	2.711	
nm	pure	1% dye	2% dye	nm	pure	1% dye	2% dye	
424.8	6.616	8.98	8.599	407.6	6.206	7.214	6.473	
424.4	6.539	8.916	8.659	407.2	6.318	7.232	6.5	
424	6.488	8.853	8.704	406.8	6.43	7.282	6.527	
423.6	6.436	8.776	8.734	406.4	6.545	7.316	6.538	
423.2	6.409	8.715	8.749	406	6.682	7.336	6.566	
422.8	6.364	8.639	8.746	405.6	6.841	7.339	6.607	
422.4	6.297	8.579	8.729	405.2	7.022	7.366	6.629	
422	6.231	8.549	8.696	404.8	7.22	7.394	6.636	
421.6	6.188	8.52	8.632	404.4	7.391	7.45	6.659	
421.2	6.168	8.491	8.537	404	7.563	7.492	6.698	
420.8	6.172	8.447	8.412	403.6	7.713	7.518	6.736	
420.4	6.197	8.388	8.288	403.2	7.842	7.529	6.759	
420	6.223	8.314	8.163	402.8	7.923	7.54	6.766	
419.6	6.27	8.239	8.022	402.4	7.981	7.583	6.79	
419.2	6.362	8.197	7.867	402	8.017	7.655	6.845	
418.8	6.499	8.183	7.712	401.6	8.051	7.74	6.9	
418.4	6.679	8.153	7.588	401.2	8.084	7.808	6.97	
418	6.837	8.107	7.482	400.8	8.118	7.861	7.057	
417.6	6.996	8.045	7.345	400.4	8.122	7.946	7.125	
417.2	7.131	7.984	7.207	400	8.105	8.063	7.193	
416.8	7.266	7.922	7.098	399.6	8.065	8.18	7.294	
416.4	7.4	7.861	7.02	399.2	8.025	8.332	7.415	
416	7.512	7.799	6.925	398.8	7.988	8.469	7.521	
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415.6	7.58	7.722	6.815	398.4	8	8.589	7.626
415.2	7.625	7.629	6.704	398	8.032	8.71	7.732
414.8	7.624	7.549	6.625	397.6	8.064	8.831	7.821
414.4	7.601	7.501	6.563	397.2	8.096	8.951	7.931
414	7.557	7.469	6.484	396.8	8.154	9.06	8.077
413.6	7.491	7.409	6.406	396.4	8.211	9.168	8.223
413.2	7.385	7.349	6.359	396	8.295	9.263	8.354
412.8	7.256	7.274	6.329	395.6	8.379	9.357	8.468
412.4	7.106	7.23	6.313	395.2	8.437	9.451	8.583
412	6.954	7.216	6.298	394.8	8.495	9.545	8.698
411.6	6.803	7.232	6.298	394.4	8.606	9.621	8.813
411.2	6.652	7.247	6.33	394	8.69	9.681	8.912
410.8	6.502	7.232	6.375	393.6	8.775	9.726	8.995
410.4	6.351	7.187	6.404	393.2	8.839	9.757	9.079
410	6.201	7.159	6.417	392.8	8.881	9.788	9.147
409.6	6.095	7.164	6.43	392.4	8.875	9.819	9.2
409.2	6.032	7.2	6.457	392	8.876	9.834	9.29
408.8	6.014	7.22	6.484	391.6	8.877	9.833	9.38
408.4	6.041	7.224	6.497	391.2	8.878	9.832	9.455
nm	pure	1% dye	2% dye	nm	pure	1% dye	2% dye
235.6	7.165	7.694	6.728	218.4	7.218	8.304	8.657
235.2	6.922	7.727	6.823	218	7.154	8.237	8.701
234.8	6.679	7.746	6.92	217.6	7.068	8.138	8.73
234.4	6.437	7.78	7.031	217.2	6.939	8.038	8.759
234	6.194	7.848	7.127	216.8	6.787	7.972	8.789
233.6	5.974	7.931	7.206	216.4	6.634	7.935	8.818
233.2	5.775	8.031	7.317	216	6.457	7.898	8.847
232.8	5.599	8.116	7.46	215.6	6.302	7.86	8.876
232.4	5.424	8.183	7.636	215.2	6.126	7.808	8.905
232	5.249	8.251	7.795	214.8	5.929	7.725	8.917
231.6	5.097	8.305	7.938	214.4	5.73	7.642	8.913
231.2	4.943	8.358	8.067	214	5.574	7.559	8.909
230.8	4.79	8.412	8.195	213.6	5.396	7.509	8.905
230.4	4.68	8.493	8.323	213.2	5.241	7.487	8.934
230	4.592	8.575	8.452	212.8	5.108	7.499	8.982
229.6	4.504	8.641	8.549	212.4	4.997	7.494	9.014
229.2	4.438	8.691	8.63	212	4.908	7.49	9.047
228.8	4.371	8.725	8.696	211.6	4.841	7.469	9.079
228.4	4.327	8.759	8.762	211.2	4.774	7.433	9.096
228	4.327	8.778	8.812	210.8	4.751	7.382	9.097
227.6	4.349	8.781	8.846	210.4	4.749	7.363	9.082
227.2	4.392	8.784	8.849	210	4.772	7.378	9.068
226.8	4.456	8.787	8.835	209.6	4.794	7.425	9.053
226.4	4.544	8.819	8.838	209.2	4.838	7.507	9.021
226	4.631	8.865	8.855	208.8	4.925	7.589	8.99
225.6	4.739	8.895	8.873	208.4	5.035	7.654	8.958

225.2	4.872	8.893	8.89	208	5.123	7.704	8.926
224.8	5.027	8.86	8.892	207.6	5.233	7.732	8.872
224.4	5.183	8.827	8.877	207.2	5.366	7.846	8.905
224	5.339	8.794	8.846	206.8	5.498	7.976	8.938
223.6	5.539	8.761	8.816	206.4	5.597	8.121	8.989
223.2	5.761	8.756	8.785	206	5.86	8.266	9.055
222.8	6.008	8.751	8.754	205.6	6.102	8.411	9.121
222.4	6.257	8.747	8.723	205.2	6.365	8.54	8.832
222	6.484	8.712	8.692	204.8	6.273	8.654	8.881
221.6	6.667	8.663	8.645	204.4	6.535	8.752	8.914
221.2	6.828	8.597	8.611	204	6.475	8.866	8.962
220.8	6.967	8.531	8.578	203.6	6.262	8.504	8.534
220.4	7.085	8.465	8.544	203.2	7.2	9.999	9.999
220	7.179	8.4	8.51	202.8	7.22	9.999	9.999
219.6	7.251	8.365	8.507	202.4	6.553	9.999	9.999
219.2	7.277	8.361	8.536	202	9.999	9.999	9.999

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