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***FABRICATION OF SOLAR CELLS USING SPRAY PYROLYSIS
TECHNIQUE***

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إهداء
وَآخِرُ دَعْوَاهُمْ أَنِ الْحَمْدُ لِلَّهِ رَبِّ الْعَالَمِينَ

لم تكن الرحلة قصيرة، ولا الطريق سهلاً دائماً، لكن الحمد لله الذي أتم هذا الحلم وبلغني هذه اللحظة التي انتظرتها طويلاً.

إلى من كانت سندي الأول، ومصدر قوتي وثقتي بنفسي، إلى من كانت دعواتها ترافقني في كل خطوة، ومن جعل الله الجنة تحت قدميها، إلى من منحني الحب والقوة والطمأنينة دون مقابل...

أمي الحبيبة.

إلى من تعب لأجل راحتي ونجاحي، وكان دعمه صامتاً لكنه عظيم الأثر في قلبي و من كان وجوده دائماً أماناً أستند إليه ...

أبي الغالي.

إلى أخي الصغير، قطعة القلب وبهجة البيت، أسأل الله أن يقرّ أعيننا بك، وأن نراك في أعلى المراتب، كما تمنينا لك دائماً.

إلى الأصدقاء الأوفياء، رفقاء الطريق والسنين، إلى من جعلوا الأيام أخفّ واللحظات أجمل، وكانوا خير سند في كل لحظة تعب وفرح...

والحمد لله على ما وهبني وما أعانني عليه.

بيعه وصال



في لحظة لطالما حلمت بها، لحظة التتويج بثمرة تعبٍ وتعبٍ طويل، لا يسعني إلا أن أرفع أسمى عبارات الشكر لله عزَّ وجلَّ، ثم لكل من كان سندًا حقيقيًا في رحلتي.

إلى أبي الحبيب،
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كنت الصخرة التي استندت عليها، والقلب الذي دعمني دون شروط... شكرًا لك بحجم السماء.

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كل حرف تعلمته، وكل خطوة خطوتها، كان لك فيها أثرٌ لا يُنسى... أحبك أكثر مما تقول الكلمات.

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General Introduction

Today, the global energy sector is facing a critical turning point. The rapid depletion of fossil fuels, combined with the accelerating impacts of climate change, has made it urgent for us to find new solutions. To protect our environment and ensure long-term energy security, we must shift toward clean, inexhaustible sources of power [1]. Among the choices available, solar photovoltaic (PV) technology stands out as one of the best pathways forward. By directly converting sunlight into electricity using semiconductor materials, solar cells give us a clean way to cut down on harmful emissions and meet the world's growing energy needs [2].

Algeria is in an exceptional position to lead this transition because of its geography and vast desert landscapes. The Algerian Sahara covers about 86% of the country, enjoying between 3,000 and 3,900 hours of sunshine every single year. In these southern regions, solar radiation reaches up to 2,650 kWh/m²/year, representing an immense theoretical potential of 170 TWh annually [3]. This abundance of sunlight highlights how valuable it is to research, develop, and optimize solar energy systems right here within the Algerian academic community.

While traditional silicon wafer solar cells dominate the current commercial market, they require a lot of energy and are expensive to manufacture. To make solar energy more affordable and versatile, researchers have turned to thin-film solar cells. These devices use very little material, with active layers ranging from just a few nanometers to a couple of micrometers thick [4]. Among the materials used for thin films, transition metal oxides have gained a lot of attention. They are highly abundant in nature, non-toxic, structurally stable, and incredibly cheap to produce [5].

This Master's thesis focuses on creating and optimizing a self-assembled p-n heterojunction thin-film solar cell made of Copper Oxide and Zinc Oxide. Within this device, the CuO layer serves as a narrow-bandgap (1.2 – 1.7 eV) p-type semiconductor designed to capture a broad spectrum of visible light. On top of it, the ZnO layer acts as a wide-bandgap (≈ 3.3 eV) n-type semiconductor. This forms a highly transparent window that lets visible light pass straight through to the active electrical interface while safely blocking harmful high-energy ultraviolet radiation. To keep the fabrication process scalable and low-cost, we chose the Chemical Spray Pyrolysis (CSP) technique to deposit these films from acetate solutions at a controlled substrate temperature of $T_s=300$ C.

To present our work clearly, this thesis is divided into three main chapters:

- **Chapter I: General Overview of Solar Cells** explores the history of photovoltaics, reviews global energy challenges, details Algeria's solar energy wealth, and explains how different types of solar cells work.
- **Chapter II: Materials and Experimental Methods** detail how thin films grow, introduces the science behind Chemical Spray Pyrolysis, and guides you through

our laboratory steps for preparing samples, mixing solutions, and testing materials (using XRD, UV-Vis, and SEM/EDX).

- **Chapter III: Results and Discussion** present and interprets our experimental data. It connects the optical and structural features of our single layers to the hidden chemistry happening at the interface of our integrated p-CuO}/n-ZnO solar cell.

Chapter I: General overview of solar cell

I.1 Introduction

The energy sector is changing a lot because we are running out of fuels and climate change is getting worse. Solar energy is one of the ways to get the energy we need. We can make electricity from sunlight using special materials. This is a way to reduce the bad things we put into the air and make sure we have energy for a long time.

To understand how we can make solar devices we need to look at how solar energy has changed over time. We will look at how people used energy a long time ago and how it is used now. We will also look at how much solar energy Algeria can produce and how solar cells work. Then we will talk about what we want to achieve with this study. We want to make film solar cells that work better and do not cost too much. Solar energy is a part of this and we will focus on solar cells to make this happen. The goal is to make cells that are good for the environment and good for our wallets and solar energy is the key, to this [6].

I.2 History of Solar Energy and Solar Cells

I.2.1 Early Use of Solar Energy

People have been using energy for a very long time. We know that around the century B.C. People used sunlight to start fires with special materials that worked like magnifying glasses. A little later by the 3rd century B.C., the Greeks and Romans used mirrors to light torches for events. These mirrors were called burning mirrors. The Chinese also used sunlight in a way around 20 A.D. People did not just use solar energy for technology they also used it when they built things. For example, the old Roman bathhouses had rooms that faced the south so they could get much sunlight as possible. This helped keep the rooms warm. Years later the Anasazi people, who are the ancestors of the Pueblo Native Americans built their homes on cliffs that faced the south. They did this so they could stay warm during the winter months. During the 19th centuries people tried to use sunlight to power things like ovens that they could take with them on long trips. They even tried to make powered steamboats. Solar energy has been an idea, for centuries, the use of

energy is not a new concept it has deep historical roots and people have been using solar energy for thousands of years [6].

I.2.2 Discovery of the Photovoltaic Effect

The discovery of the photovoltaic effect dates back to the nineteenth century, when the scientist Edmond Becquerel, in 1839, studied the effect of light on certain materials and solutions, as well as the electrical current generated from them. Later, in 1877, the scientists Willoughby Smith and Adams introduced the concept of photoconductivity for the first time. In 1883, Charles Fritts developed the first solar cell using selenium (Se), demonstrating the potential of generating electricity from sunlight. The first solar cell with an efficiency of about 1% was produced in 1941. This was followed by the development of a solar cell with an efficiency of approximately 6% by Bell Laboratories in the United States, which was initially used in space applications. Subsequently, solar cells made from cadmium sulphide and copper sulphide materials were developed, later known as thin-film solar cells. Since then, research in physics, engineering, and semiconductor science has accelerated significantly, particularly the study of photoelectric interactions, leading to major advancements in photovoltaic technology, with power outputs ranging from milliwatts to kilowatts [7].

I.2.3 Milestones in Solar Cell Development

Solar cell technology has come a long way, thanks to several key breakthroughs that have made it more efficient and useful. A significant leap forward happened in the 1950s when researchers shifted to silicon-based solar cells. In 1954, Daryl Chapin, Calvin Fuller, and Gerald Pearson at Bell Laboratories developed the first practical silicon solar cell, which managed about 6% efficiency. This marked the beginning of what we now know as modern photovoltaic technology, opening doors for real-world applications. The intense competition of the space race during the late 1950s and 1960s further spurred innovation. Solar cells began powering satellites, with Vanguard 1 launching in 1958 as a notable example. This showcased their ability to function reliably even in tough

conditions. By the early 1960s, advancements in materials and production techniques bumped efficiency up to around 15%. In the decades that followed, experts focused on making solar technology even better and cheaper. New strategies like anti-reflective coatings and advanced designs led to efficiencies surpassing 20% by the 1980s. Thin-film technologies also emerged, paving the way for flexible and more affordable solar options.

Today, we're seeing solar cells achieving over 25% efficiency in labs. As they become more widely produced and costs keep dropping, solar energy is rapidly becoming one of the most popular renewable energy sources across the globe [8].

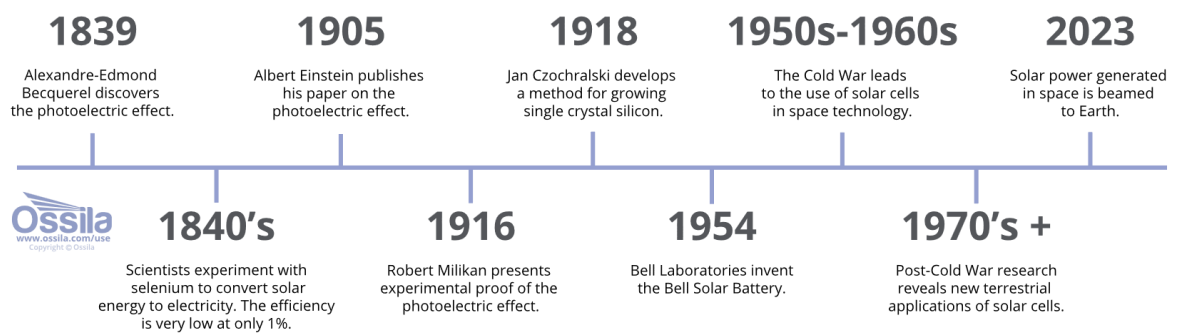


Fig I.1 History of Solar Cells [9]

I.3 Background and Context

I.3.1 Global Energy Challenges

There are some interrelated problems with global energy systems both at national and international levels. The absence of proper global governance in this respect leads to ineffective collaboration of states in terms of energy policies. There are also some distortions in the functioning of energy markets, since there is government intervention in terms of supply and demand, resulting in market failures. Investment in future energy supply is not sufficient enough, creating additional price instability and posing threats to energy security in the future. Moreover, states focus on their national interests, such as energy independence, rather than on global governance. The lack of regulation is another

important problem, since environmental problems are not effectively addressed. In particular, environmental impact of activities related to energy generation on the international level are ignored. It includes climate change, pollution of the atmosphere, emissions of dangerous substances, like mercury, as well as acid rain. Finally, the issue of energy is complex, touching upon security, environmental issues, human rights and many other aspects. Energy issues at the international level are characterized by incoherence and fragmentation [10]

I.3.2 Importance of Renewable Energy

Renewable energy is a critical factor for sustainable development, and it is one of the ways to overcome various energy challenges in the world. Renewable energy is characterized by its abundance and the fact that it cannot be depleted since it is an inexhaustible source of power that will last for many years. Renewable energy sources like solar, wind, hydro, biomass, and geothermal emit very little greenhouse gases and therefore help in combating environmental pollution and global warming. In recent times, there have been huge reductions in the supply of fossil fuel due to their negative impact on the environment. The use of renewable energy would help to eliminate the harmful effects associated with the combustion of fossil fuel and protect the environment from further degradation. Besides, renewable energy would ensure greater energy security because it does not rely on foreign energy and is cheaper than other sources [11]. Thus, renewable energy is vital for sustainable energy development.

I.3.3 Solar Energy Potential in Algeria

One of the major advantages that Algeria enjoys is its rich solar energy potential due to the geographical position of the country, as well as the huge number of deserts. The country is characterized by a high level of solar irradiation, especially in the southern part of the state, as it can reach 2263 kWh/m²/year.

The Algerian Sahara Desert covers approximately 86% of the Algerian territory, where there is a good amount of sunshine that lasts for 3000 to 3900 hours each year,

accompanied by high solar radiation, which reaches 2650 kWh/m²/year. Hence, it can provide excellent opportunities for generating energy using PV and CSP systems.

Overall, the solar energy potential of Algeria is considered to be approximately 170 TWh each year, which makes it one of the best countries in the MENA region in terms of the amount of solar energy available in this country. It should be noted that in the north of the country, including Algiers, there is also an excellent sunshine duration of 2800+ hours [12].



Fig I.2 Ain-El-Ibel - Djelfa - Solar Plants - 53MW – Algeria [13]

I.4 Solar Cell Fundamentals

I.4.1 Photovoltaic Effect Principle

Photovoltaic Effect is the basic mechanism through which the conversion of energy from solar cells takes place. This happens due to the impact of photons on a semiconductor material such as silicon, giving energy to electrons and making them mobile.

$$E=h\nu\ldots\ldots\ldots (I.1)$$

If there is enough photon energy, the electrons will get excited and will make the transition from the valence to the conduction band and create electron-hole pairs. The separation of the charge carriers is done through the electric field that exists within the p–n junction of the solar cell. As a result, electrons move throughout the external circuit creating electric current [14,15].

In brief, the mechanism can be explained as follows:

- Light absorption (photons)
- Formation of electron-hole pairs
- Charges separation with the help of the electric field
- Electron flow resulting in electrical energy production

I.4.2 Types of Solar Cells

There are different ways that solar cells can be classified depending on the manufacturing process and materials used. These classes include:

a) **Monocrystalline Silicon Solar Cells**

Monocrystalline cells contain single-crystal structures, providing efficient energy conversion rates (15-22%) and longevity. Such cells have uniform coloration and are somewhat costly to manufacture.[16]

b) **Polycrystalline Silicon Solar Cells**

Such cells consist of several silicon crystals, reducing the costs of fabrication but lowering the conversion efficiency (13-18%). Usually, polycrystalline cells have a distinctive blue hue [16].

c) **Thin-Film Solar Cells**

Thin-film cells use deposition processes to apply thin films of photovoltaic materials (e.g., amorphous silicon, CdTe, or CIGS) onto substrates. They are lightweight, flexible, and affordable, although their conversion efficiencies are inferior to those of silicon cells [17].

d) **Perovskite Solar Cells**

The newest solar cells, made out of perovskite-structured materials. They boast impressive efficiency rates and affordable production, but the stability issues remain unsolved [17,18].

e) Organic (Polymer) Solar Cells

Such cells consist of organic materials and have high flexibility and light weight. Nevertheless, their energy conversion efficiency is lower than that of traditional cells [18].

I.4.2.1 Silicon-Based

Silicon-based solar cells are the most commonly used photovoltaic technology for converting sunlight into electricity. They are made from silicon, a semiconductor material that is highly effective due to its ability to generate electric charge when exposed to light. When sunlight strikes the silicon, it excites electrons and creates electron–hole pairs, which are then separated by the internal electric field of the p–n junction, producing an electric current. Silicon solar cells are mainly divided into monocrystalline and polycrystalline types, where monocrystalline cells offer higher efficiency while polycrystalline cells are more cost-effective. Overall, silicon-based solar cells are widely used because they are reliable, durable, and have a long operational lifespan, although their production can be relatively expensive and energy-intensive [19,20].



Fig I.3 Silicon-based solar cells [21]

I.4.2.2 Thin-Film

A thin-film solar cell is a type of photovoltaic cell where very thin films of semiconductor materials are layered onto a substrate such as glass, plastic, or even metal. This includes the use of materials like amorphous silicon, CdTe (cadmium telluride), or CIGS (copper indium gallium selenide). This method is much cheaper than that used for conventional silicon solar cells because it uses far less material, which makes it possible to develop cells that are lighter and more flexible, making them ideal for many applications. They, however, tend to be less efficient and have a relatively short lifespan due to the particular material used [22,23]

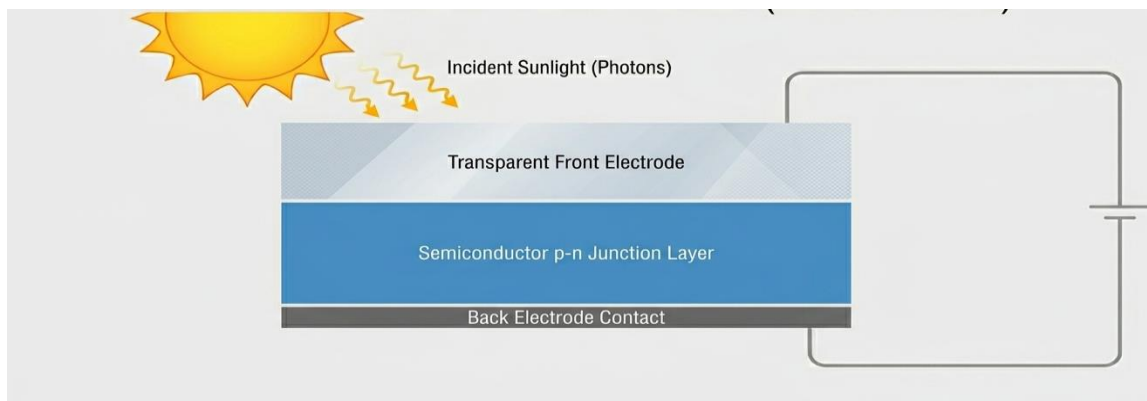


Fig I.4 A thin-film solar cell

I.4.2.3 Emerging Solar Cell

Emerging solar cells can be defined as modern solar cells which are evolving and advancing from conventional solar cells based on silicon wafers. Such technologies include, but are not limited to, perovskite solar cells, organic solar cells, dye-sensitized solar cells, and tandem solar cells. The potential of perovskite solar cells lies in their ability to absorb more light and their rising efficiency rates, despite their inability to last as long as traditional solar cells. Organic solar cells can be considered lightweight and flexible; therefore, they can be used to develop portable or wearable electronics, but their efficiency is low, and their lifespan is short. On the other hand,

tandem solar cells consist of two or more materials, allowing them to absorb a broader spectrum of sunlight and thus increase their efficiency rates [24].

I.5 Objectives of the Study

I.5.1 Main Objective

Mainly, the goal of this research work is the development and optimization of thin film solar cells by adopting the spray pyrolysis deposition method, which would help increase their photovoltaic properties. This includes improving their physical structure, optical absorptivity, and electrical properties without losing affordability and scalability of the process employed.

I.5.2 Specific Objectives

The following objectives are set to achieve the primary aim of this work:

- a) **Preparation of Thin Films:** The synthesis of thin films of semiconductor material via the use of spray pyrolysis in an optimized experimental setup.
- b) **Optimization of Film Properties:** The effect of important parameters like substrate temperature, precursor concentration, spray rate, and distance between nozzle and substrate on the properties of thin films will be investigated.
- c) **Structural Properties:** Investigation into the structural properties, which include the structure, phases present, and grain sizes of the thin films will be done via X-ray diffraction.
- d) **Film Morphology:** Investigation of surface morphology of the thin films and uniformity in deposition, and other morphological properties like surface roughness and distribution of grains will be carried out.
- e) **Film Optical Properties:** Investigation into the optical properties of the films, which will include absorbance, transmittance, and optical band gap energy, will be carried out via UV-Vis spectroscopy.

- f) **Film Electrical Properties:** Study into the electrical properties of the films, which include conductivity, carrier concentration, and mobility, for assessment of the applicability of the films for photovoltaics.
- g) **Photovoltaic Properties:** Evaluation of the photovoltaic behaviour of the fabricated solar cells through parameters like current density, voltage, and efficiency.
- h) **Comparative Analysis:** For the purpose of comparing the results obtained in the laboratory with those from literature for verifying the effectiveness of the selected materials and technique used.
- i) **Contribution to the Local Research Environment:** To study the possibility of using solar cells in areas where there is high irradiation of sunlight, like Algeria.

I.6 Conclusion

This chapter presented a general overview of solar energy and solar cell technology, establishing the theoretical and contextual foundation for this study.

It began with a historical perspective on the use of solar energy, showing how ancient civilizations utilized sunlight for practical purposes such as heating and ignition. These early applications demonstrate that solar energy has been used for thousands of years, long before the development of modern technologies.

The chapter then discussed the scientific development of solar cells, starting with the discovery of the photovoltaic effect in the nineteenth century. This discovery marked a major step toward converting sunlight into electricity. Later advancements, particularly the development of silicon-based solar cells in the mid-twentieth century, enabled practical applications and significantly improved efficiency, leading to the widespread use of photovoltaic systems.

In addition, global energy challenges were addressed, including energy insecurity, environmental pollution, and the depletion of fossil fuel resources. In this context, renewable energy sources were presented as a sustainable solution, with solar energy being one of the most promising options due to its abundance and low environmental impact.

Special attention was given to the solar energy potential of Algeria. The country benefits from high solar irradiation and long sunshine duration, especially in the Sahara region, making it highly suitable for photovoltaic energy production. This highlights the importance of developing solar technologies at the national level.

The fundamental principles of solar cell operation were also explained, focusing on the photovoltaic effect as the mechanism responsible for converting light into electrical energy. The processes of photon absorption, generation of electron–hole pairs, and charge separation within the semiconductor were described.

Furthermore, different types of solar cells were reviewed, including silicon-based, thin-film, and emerging technologies. Each type was analyzed in terms of efficiency, cost, and practical applications. Particular emphasis was placed on thin-film solar cells due to their advantages, such as reduced material consumption, flexibility, and potential for low-cost production.

Finally, the objectives of the study were clearly defined, outlining the main goal and the specific tasks required to achieve it. These objectives provide a clear direction for the experimental work presented in the next chapter.

Overall, this chapter provides a solid background for understanding the importance of solar energy and the development of photovoltaic technologies. The next chapter will focus on the materials and experimental methods used in this research.

Chapter II: Materials and Experimental Methods

II.1 Introduction

Thin film technology is regarded as one of the major inventions for the progress of technology, especially semiconductor technology. New ways of production and manufacturing processes have developed due to the advancement of science, allowing more accurate determination and unification of layer thicknesses. These developments have made it possible to discover certain characteristics of the material [25].

In this chapter, we are going to learn about thin films, manufacturing processes of thin film and deposition process, as well as physical methods of investigation of such thin film properties.

II.2 Thin-Film Solar Cells

Thin films are used to describe one or several layers of a material whose thickness does not exceed one micrometer or a few nanometers [26]. They are formed as a result of the condensation of atoms or molecules on a substrate (solid base), which can be made of glass, silicon, or aluminum depending on the nature of the study [27]. The fundamental difference between materials in the bulk (solid state) and those in the form of thin films lies in their physical properties. In bulk materials, surfaces (boundaries) generally play a minor role in determining physical properties. However, in thin films, the opposite is true: surface effects dominate the physical properties. Another essential feature of thin films is that they are almost always deposited on a substrate, regardless of the preparation method. Therefore, special attention must be given to the nature and structure of the substrate, as it significantly influences the structural properties of the thin films [28].

II.2.1 Advantages and Applications

Thin film solar cells have many benefits which make them suitable for different uses. They are lightweight and flexible, thus able to be used in curved surfaces and building materials. Their production is less costly than silicon cells due to their simpler production method. Besides, they operate effectively in low light and high temperatures, and might experience less degradation with time. As such, the thin-film technology is very flexible,

making it applicable in diverse areas, including building-integrated photovoltaics, aerospace, transportation, and portable equipment [29,30,31].

Benefits of Thin Film Solar Cells:

- ✓ Lightweight and flexible [29]
- ✓ Less costly to produce
- ✓ Effective at low light and high temperature levels [30]
- ✓ May degrade less with time

Uses of Thin Film Solar Cells:

- ✓ Building integrated photovoltaics (BIPV): roof and window panels, solar glass [31]
- ✓ Space technology: satellites and aerospace equipment
- ✓ Automotive and maritime application: automobiles, public transport vehicles, ships
- ✓ Personal use: solar chargers, portable solar panel sets

II.2.2 Materials Commonly Used

Transparent Conductive Oxides (TCOs): Materials such as Tin Oxide (SnO_2), Fluorine-doped Tin Oxide (FTO), and Indium Tin Oxide (ITO) [32].

Metal Oxides: Including Titanium Dioxide (TiO_2), Nickel Oxide (NiO), and Iron Oxide (Fe_2O_3) [33].

Binary and Ternary Compounds: Various metal-based salts used for sensors and photovoltaic buffer layers.

II.2.3 Rationale for Choosing Zinc and Copper Oxides

Electrical polarity (n-p pair):

ZnO is an n-type semiconductor, and copper oxides display p-type conductivity, thus forming p-n junctions that could be used for optoelectronic applications [34,31].

Band gap complementarity:

Due to its wide bandgap (~ 3.37 eV), ZnO shows transparency, whereas CuO and Cu₂O possess narrow gaps (~ 1.2 - 2.1 eV), which allows effective light harvesting [35,31].

Phasic flexibility:

Copper oxides can crystallize in distinct phases (CuO and Cu₂O) that could be regulated through controlling the growth parameters, namely the temperature of deposition and type of precursors [31].

Potential for thin-film applications:

Combination of ZnO and CuO is intensively explored in the context of thin films used as heterostructures in photovoltaics and sensing applications [36].

Sustainability and economic feasibility:

As mentioned above, zinc and copper are cheap and ecologically friendly materials, which makes their use preferable from these aspects [26,28].

Table II.1 Properties of CuO and ZnO

Propriétés	Valeur
Masse molaire (g/mol)	79.55
Solubilité dans l'eau (mg/L)	Insoluble
Point de fusion (C°)	1326
Enthalpie de formation à 298 K	-157.3 kJ/mol
Entropie S ₀ (JK ⁻¹ mol ⁻¹)	42.6
Capacité calorifique Cp (Jmol ⁻¹ K ⁻¹)	42.3
Conductivité thermique (Wcm ⁻¹ K ⁻¹)	33 W/m-K (scaled or correct range for bulk)

Propriétés	Valeur
Masse molaire (g/mol)	81.37
Solubilité dans l'eau (mg/L)	Soluble [2]
Point de fusion (C°)	1975[3]
Enthalpie de formation à 298 K	-348.3
Entropie S ₀ (JK ⁻¹ mol ⁻¹)	43.6
Capacité calorifique Cp (Jmol ⁻¹ K ⁻¹)	40.3[4]44.7 à 300K ou 50.6 à 800 K [5]
Conductivité thermique (Wcm ⁻¹ K ⁻¹)	0.54[5]

II.3 Classification of Thin-Film Deposition Techniques

Various techniques for thin film deposition have been classified by nature and state of the deposition process and precursor material.

II.3.1. Physical Vapor Deposition (PVD)

The PVD method requires a physical change of a material from solid/liquid to gaseous state and back, resulting in the deposition of material on the substrate.

- a) **Thermal Evaporation:** The source material is heated in a high vacuum environment causing evaporation [37].
- b) **Sputtering:** High energy ions (typically Argon) cause ejection of the atoms of the target material [38]
- c) **Pulsed Laser Deposition (PLD):** High power laser causes vaporization of the target material creating a plasma plume [39]

II.3.2. Solution-Based Chemistry Methods

- **Sol Gel Process:**

Based on the process of transformation of the system from liquid phase to gel phase [40,41].

- **Spin-Coating:**

Deposition by using the centrifugal force of spinning [42].

- **Dip-Coating:**

Deposition by dipping the substrate in the solution with a certain rate of deposition [43]

- **Chemical Bath Deposition (CBD):**

Process that is used for precipitation of films from a chemical reaction in a liquid bath [44,45].

- **Chemical Spray Pyrolysis (CSP):**

Atomization process followed by the thermal decomposition of the precursor solution [46]

II.4 Chemical Spray Pyrolysis (CSP)

The spray pyrolysis technique is a chemical method used for the production of thin films. It is considered a pneumatic variant of chemical vapor deposition (CVD). This technique is characterized by its simplicity and relative cost-effectiveness, particularly in terms of equipment requirements. It is widely used for depositing various materials, including transparent conducting oxides like zinc oxide (ZnO) and nickel oxide (NiO) [47].

II.4.1 Principle of the Technique

The spray pyrolysis technique is a chemical deposition method characterized as a pneumatic variant of chemical vapor deposition (CVD). The fundamental principle of this method involves the pulverization of a precursor solution into fine droplets using an atomizer under the pressure of a carrier gas. These droplets are then directed onto a heated substrate [47,48]

II.4.2 Advantages and Limitations

a) Advantages:

Low Cost: Eliminates the need for expensive high-vacuum systems, significantly reducing capital investment [49].

Simple Doping: Electrical and optical properties are easily adjusted by adding dopants directly to the liquid precursor [50].

Scalability: Highly suitable for large-area industrial coating, vital for the Algerian solar sector [51].

Strong Adhesion: High-temperature reactions ensure strong chemical bonding between the ZnO film and the substrate [51].

b) Limitations:

- ✓ **Surface Roughness:** Films often show higher roughness compared to vacuum-based sputtering methods [52].
- ✓ **Heat Constraints:** Restricted to substrates that can withstand temperatures up to 500 C [53].
- ✓ **Precision Limits:** Achieving uniform layers below 10 nm is more difficult than with Atomic Layer Deposition [54].

II.5 Spray Pyrolysis System Components

a) Sprayer (Nozzle):

The sprayer is a pneumatic device made of chemically resistant plastic (such as Lucite), capable of withstanding the chemical effects of the precursor solutions. The lower end of the nozzle contains two channels: one for the solution and the other for the gas, allowing efficient atomization of the solution onto the substrates [55].

b) Heating System:

The heating system consists of a substrate holder placed a few millimetres above a resistive heating element embedded in a block and mounted in a metallic container. Its regulation system minimizes temperature fluctuations caused by the sprayed solution and the carrier gas flow. Its main function is to heat the substrate to the required temperature for compound formation [55].

c) Thermocouple (Temperature Control):

A Nickel-Chromium thermocouple attached to the heating plate and connected to a direct-reading thermometer is used to control the reaction temperature. It also allows relative monitoring of the substrate temperature during the spray process [55].

d) Gas Generator:

The gas system transports the sprayed solution from the nozzle to the substrate. The gas flow has two main advantages: it can be precisely controlled, and the

gases used may also participate as reactive species in the composition of the deposited material [55].

II.6 Experimental Procedure (Data Collection)

II.6.1 Substrate Cleaning

The cleaning of substrates is an essential step to ensure good adhesion, uniform film growth, and to eliminate any surface contamination that may affect the properties of the deposited thin films.

The cleaning procedure was carried out as follows:

- Soda-lime glass substrates were cut into small squares of approximately 2.54×2.54 cm.
- The substrates were washed with detergent and rinsed with running water to remove dust and surface particles.
- The samples were then immersed in ethanol (96–99%) for 5 minutes to remove organic impurities such as grease and oil.
- After that, the substrates were rinsed thoroughly with deionized water to eliminate any remaining residues.
- The substrates were dried using lint-free optical paper.
- Finally, the cleaned substrates were immediately placed on the hot plate to avoid re-contamination from the laboratory environment.

All substrates were handled using tweezers throughout the process to maintain surface cleanliness.

II.6.3 Thin Film Deposition by Spray Pyrolysis

The thin films were deposited using the chemical spray pyrolysis technique, in which a precursor solution is atomized into fine droplets and sprayed onto a heated substrate.

The deposition process was carried out as follows:

- The cleaned substrates were placed on a heating plate maintained at approximately 300°C, as preliminary experiments conducted at lower temperatures and at 450°C resulted in unsatisfactory deposition outcomes.
- The precursor solution was introduced into a pneumatic spray nozzle system.
- Compressed air was used to atomize the solution into fine aerosol droplets.
- The nozzle-to-substrate distance was maintained between 15 and 25 cm to ensure uniform deposition.
- The solution was sprayed in a pulsed mode to avoid excessive cooling of the substrate surface.
- Two deposition conditions were used by varying the solution volume (20 mL and 30 mL) to obtain films with different thicknesses.
- After deposition, the films were allowed to cool naturally on the heating plate until room temperature.
- For multilayer structures, a copper-based layer was first deposited, followed by a zinc oxide layer, forming a ZnO/CuO heterostructure.

II.7 Experimental Tools and Materials

The experimental work carried out in this study was based on standard laboratory equipment and high-purity chemical reagents suitable for thin film deposition using the spray pyrolysis technique

II.7.1 Chemicals and Precursors

- Zinc acetate dihydrate [$Zn(CH_3COO)_2 \cdot 2H_2O$], used as the zinc precursor.
- Copper (II) chloride [$CuCl_2$], used as the copper precursor.
- Ethanol (96–99% purity), used as the solvent for precursor solution preparation

II.7.2 Laboratory Equipment

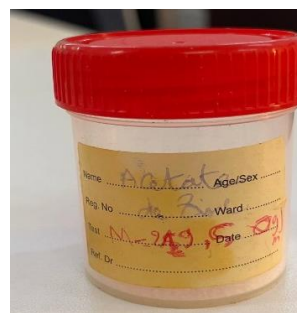
- **Magnetic stirrer:** Used to ensure homogeneous mixing of precursor solutions.
- **Digital balance:** Used for accurate weighing of chemical precursors.
- **Heating plate (hot plate):** Used to maintain the substrate temperature during deposition (around 300°C).
- **Pneumatic spray atomizer (spray nozzle):** Used to convert the precursor solution into fine aerosol droplets.
- **Temperature controller / thermometer:** Used to monitor and regulate substrate temperature during deposition.
- **Soda-lime glass substrates:** Used as deposition surfaces for thin film growth.
- **Glassware (beakers, graduated cylinders, pipettes):** Used for accurate solution preparation and volume measurement.



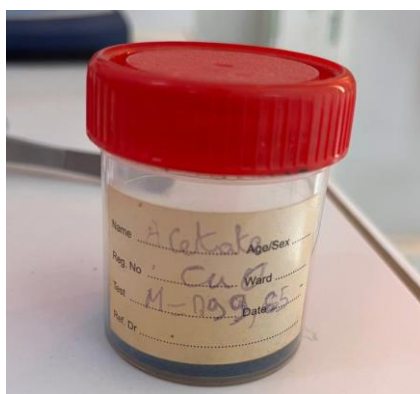
a. Magnetic stirrer



b. glass substrates



c. Zinc acetate



d. Copper acetate



e. Ethanol

Fig II.1 Laboratory Equipment

II.8 Control of Deposition Parameters

The precise regulation of deposition variables is fundamental to achieving high-quality semiconductor layers. These parameters directly influence the light propagation and structural density of the deposited films.

Substrate Temperature (T_s): This parameter is the primary driver of the pyrolytic reaction. Maintaining an optimal temperature ensures the complete decomposition of the precursor and promotes the formation of a well-defined crystalline lattice. For ZnO, this thermal control is vital to achieving the desired optical and structural properties [56].

Spray Rate: The flow of the precursor solution must be carefully regulated to prevent excessive cooling of the substrate surface. A steady, controlled rate ensures that the reaction remains in the

"CVD" (Chemical Vapor Deposition) regime, allowing for the growth of a dense and homogeneous film rather than a porous layer [56, 57].

Nozzle-to-Substrate Distance: This distance determines the thermal history of the droplets during their flight. Proper adjustment ensures that the droplets reach the substrate in the optimal state for decomposition. This distance directly impacts the final thickness and surface roughness, which in turn affects the electromagnetic modes and light propagation within the thin-film layer [57].

II.9 Characterization and Data Analysis Techniques

The properties of the thin films formed depend on how the precursors interact with each other and with the thermal energy during film formation. In order to analyze the integrity of the layers ZnO and CuO, the following methods of analysis were employed.

II.9.1 Structural Analysis (XRD)

X-Ray Diffraction (XRD) is the fundamental non-destructive technique used to resolve the atomic arrangement of the films. By bombarding the sample with a monochromatic X-ray beam (typically $Cu K\alpha$ radiation, $\lambda=1.5406 \text{ \AA}$, the crystalline phases are identified through Bragg's Law:

$$n \lambda = 2d \sin \theta \dots\dots\dots (II.2)$$

Phase Identification: The diffraction peaks are compared with standard JCPDS files to confirm the hexagonal wurtzite structure of ZnO or the monoclinic structure of CuO [58].

Crystallite Size: The average grain size is calculated via the Scherrer formula. A decrease in the Full Width at Half Maximum (FWHM) of the (002) peak typically indicates an improvement in crystallinity and an increase in grain size [58].

Micro-strain: The lattice strain (ϵ) induced by the thermal mismatch between the glass substrate and the film can also be calculated from the peak shifts, providing insight into the mechanical stability of the layer [50, 51].



Fig II.2 Figure of Structural Analysis (XRD) [59]

II.9.2 Optical Characterization (UV-Vis Spectroscopy)

The optical investigation is carried out in order to study the interaction of solar radiation with the semiconductor materials. Absorbance and transmittance measurements of semiconductor films are carried out using double beam spectrophotometer in the wavelength range from 300 to 1100 nm, when transmittance of more than 80% is required for application as window material in solar cells [56, 57].

Optical band gap (E_g) is obtained through Tauc plot of $(\alpha h\nu)^2$ against energy [56].

This technique provides important information about the absorption and extinction coefficient, which are significant for understanding the energy dissipation and electromagnetic modes of light propagation within the thin film [57,58].



Fig II.3 Figure of UV-Vis Spectroscopy [60]

II.9.3 Morphological Analysis (SEM/EDS)

The morphology of the films is characterized visually and chemically on a microscopic level. The Scanning Electron Microscopy (SEM) technique is used to examine the crystal surface structure and growth characteristics, thereby establishing if the spray pyrolysis process yielded a dense and continuous film or nano-structured aggregates as well as any possible surface irregularities such as "splashing" or cracks [50,52].

At the same time, the use of Energy Dispersive Spectroscopy (EDS) yields elemental maps indicating the presence of the zinc, copper, and oxygen atoms and their uniform distribution [52]. The cross-sectional scanning electron microscopy images can be used to determine the thickness of the film.



Fig II.4 Figure of Morphological Analysis (SEM/EDS) [61]

II.10 Conclusion

This chapter explained the materials, methods, and experimental procedures used to prepare and study the thin films in this work.

It started by introducing thin-film technology and its importance in modern applications, especially in solar cells. Thin films are very thin layers of material where surface effects strongly influence their properties. Compared to traditional bulk materials, they are lighter, cheaper to produce, and can be deposited on different types of surfaces, which makes them very attractive for photovoltaic devices.

The chapter then discussed the materials used in this study, mainly zinc oxide (ZnO) and copper oxides (CuO and Cu₂O). These materials were chosen because they complement each other electrically and optically, are low-cost, and environmentally friendly. This makes them suitable for forming p–n junction structures used in solar energy applications.

Different deposition techniques were also briefly reviewed, with emphasis on chemical and physical methods. Among them, spray pyrolysis was selected for this work because it is simple, inexpensive, and can be used for large-area film deposition.

The spray pyrolysis technique was then described in detail. It works by spraying a liquid precursor onto a heated surface, where it decomposes and forms a solid thin film. The main advantages of this method are its simplicity and scalability, although it can sometimes produce films with surface roughness and requires careful control of experimental conditions.

The experimental setup used in this work was also presented, including the spray system, heating unit, gas flow, and temperature control. Special attention was given to substrate cleaning, since any dust or contamination can strongly affect the quality and uniformity of the deposited films.

The preparation of the ZnO solution and the deposition process were explained step by step, highlighting the importance of parameters such as temperature, spray rate, and distance between the nozzle and the substrate. These factors directly influence the final structure and quality of the films.

Finally, the characterization techniques used to analyze the samples were introduced. X-ray diffraction was used to study the crystal structure, UV-Visible spectroscopy to investigate optical properties, and electron microscopy to observe surface morphology and composition.

Overall, this chapter gives a clear picture of how the thin films were prepared and analyzed, and it sets the stage for discussing the experimental results in the next chapter.

General Conclusion

In this Master's thesis, we successfully prepared, analysed, and optimized p-CuO n-ZnO heterojunction thin films using the highly practical and cost-effective Chemical Spray Pyrolysis (CSP) technique at $T_s = 300$ C. By utilizing simple copper acetate and zinc acetate solutions, we engineered a functional, budget-friendly solar cell architecture well-suited for sun-rich environments like Algeria. Our laboratory investigations led to several key insights:

First, optical testing using UV-Visible spectroscopy showed that our individual single layers do exactly what they were designed to do. The pure ZnO film worked beautifully as a transparent window layer, maintaining a stable visible light transmission of 22.55% and creating a sharp barrier against ultraviolet light near 380 nm. This confirms a wide direct energy bandgap (≈ 3.3 eV) that lets useful sunlight pass through while protecting the cell from UV damage. On the other hand, the individual CuO film proved to be an excellent solar absorber, showing a steady slope in light transmission, verifying its ability to capture broad solar energy across its narrow bandgap ($\approx 1.2 - 1.7$ eV)

Second, when we stacked these materials together to form a double layer, their optical behavior changed completely. The separate transparency windows disappeared, causing light transmission at 800 nm to drop down to just 1.15% for Sample 1. This was accompanied by a major increase in visible light absorption 2.31Abs, proving that the bilayer structure forms a highly effective, collaborative light trap. Sample 1 captured light much better and was structurally denser than Sample 2, showing that slight adjustments in how the precursors break down under heat can drastically reduce light loss.

Third, surface imaging via Scanning Electron Microscopy (SEM) confirmed that all our films were highly uniform, continuous, and covered the glass substrates perfectly. The top ZnO layer, modified with the 0.1g/L additive, displayed clean, hexagonal wurtzite grains (≈ 740 nm) with tiny surface pores ≈ 280 nm. Meanwhile, the underlying CuO film formed a remarkably tight, compact network of rounded tenorite grains (≈ 520 nm) with almost no visible pinholes or microscopic cracks. We also noted a clear difference between the larger grain clusters seen on the surface with the SEM and the

smaller crystalline domains calculated from our XRD charts using the Debye-Scherrer equation. This happens because individual tiny crystals naturally clump together during the spray process to lower their surface energy.

Fourth, chemical profiling via EDX confirmed that both of our oxide layers were highly pure, with no unexpected chemical pollution. The test did pick up signals for Silicon, Sodium, Calcium, and Magnesium, but these simply came from the underlying glass substrate. Because our thin films are less than a micrometer thick, the high-energy electron beam from the microscope passes right through them and interacts deeply with the supporting glass slide.

Finally, looking at the combined heterojunction interface revealed that spraying the top zinc oxide layer directly onto the hot, pre-existing copper oxide film provides a helpful *in-situ* annealing effect. This extra heat exposure gives the underlying layer more energy to crystallize better and ensures a strong mechanical grip between the two materials. Even more importantly, this thermal energy causes a small amount of atomic mixing right at the contact boundary: zinc ions migrate downward, and copper ions shift upward. This localized interdiffusion creates a thin, beautifully graded interface that smooths out energy barriers, reduces internal defects, prevents electrical leaks through pinholes, and helps separate electrical charges more efficiently.

In conclusion, our findings demonstrate that sequential chemical spray pyrolysis using cheap, non-toxic acetate precursors is a highly reliable way to build high-purity, structurally sound, and optically optimized p-CuO n-ZnO heterojunctions. This research offers a strong, practical foundation to the local Algerian scientific community, advancing our efforts to develop sustainable, affordable, and scalable thin-film solar technologies.

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ABSTRACT:

In this Master's thesis titled "Fabrication of solar cells using spray pyrolysis technique", the synthesis and characterization of inexpensive and environmentally friendly junction thin film solar cells have been studied. This thin film photovoltaics can be seen as an alternative source of energy generation from solar energy which has a great potential in Algeria. Thin films of p-type CuO and n-type ZnO were grown using the CSP technique on glass substrates at a temperature of 300 C with acetate precursor solutions. Characterizations were performed to demonstrate high window layer transmittance, good light absorbance, and good interface contact quality.

Key words: Solar cells, thin films, Spray pyrolysis, CuO, ZnO, p-n heterojunction, Photovoltaic energy, Thin-film photovoltaics, Optical properties, Renewable energy.

ملخص:

تشير هذه المذكرة بعنوان "تصنيع الخلايا الشمسية باستعمال تقنية الرش الحراري الكيميائي" للحصول على شهادة الماستر إلى عملية تصنيع ووصف الخلايا الشمسية ذات الأغشية الرقيقة المغايرة الوصلة ذات التكلفة المنخفضة والإنتاج الأخضر كبداية واعدة لتوليد الطاقة من طاقات الشمس في الجزائر. عن طريق تطبيق عملية التحلل الحراري الكيميائي بالرش (CSP)، نجحت في تحقيق عملية ترسيب الغشاء الرقيق لأكسيد النحاس الموجب CuO وأكسيد الزنك السالب ZnO من محلول الكلخات على الركيزة الزجاجية عند درجة حرارة 300C وقد تأكد من خلال عمليات التحليل البصري (UV-Vis) والحركي (XRD) والمورفولوجي (SEM/EDS) نفاذية طبقة النافذة وامتصاصها الفعال للضوء وجودة التلامس البيني.

الكلمات المفتاحية : الخلايا الشمسية، الأغشية الرقيقة، الرش الحراري الكيميائي (CSP)، أكسيد النحاس (CuO)، أكسيد الزنك (ZnO)، الوصلة غير المتجانسة (p-n Heterojunction)، الطاقة الكهروضوئية، الخصائص البصرية، الطاقات المتجددة، تحويل الطاقة الشمسية.

RESUME :

Ce mémoire de master "Fabrication de cellule solaire en utilisant spray pyrolysis technique" concerne la réalisation et les caractéristiques de cellules solaires à couches minces à hétérojonction à faible coût et écologiques, un système qui est susceptible de tirer profit du potentiel solaire massif qu'offre l'Algérie. En utilisant la méthode de pyrolyse par pulvérisation chimique (CSP), on a réussi à réaliser des couches minces d'oxydes de cuivre de nature p (CuO) et oxyde de zinc de nature n (ZnO) sur des substrats de verre à 300 C à partir de précurseurs acétates. Les essais optiques (UV-Vis), structuraux (DRX) et morphologiques (MEB/EDS) ont prouvé que la couche de fenêtre présente une grande transmittance, une grande absorption lumineuse ainsi qu'une bonne interface de contact.

Mots clés : Cellules solaires, Couches minces, Pyrolyse par pulvérisation chimique (CSP), Oxyde de cuivre (CuO), Oxyde de zinc (ZnO), Hétérojonction p-n, Énergie photovoltaïque, Propriétés optiques, Énergies renouvelables, Conversion de l'énergie solaire.