“Performance Evaluation of Dye-Sensitized Solar Cells (DSSCs) Using TiO_2 Nanoparticles: Effect of Electrolyte Type and Sintering methods”

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MASTER THESIS

“Performance Evaluation of Dye-Sensitized Solar Cells (DSSCs) Using TiO_2 Nanoparticles: Effect of Electrolyte Type and Sintering methods”

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Declarations

I hereby declare that the thesis titled “**Performance Evaluation of Dye- Sensitized Solar Cells (DSSCs) Using TiO_2 Nanoparticles: Effect of Electrolyte Type and Sintering methods”** is my original work carried out under the supervision of Prof. Teresa Gatti and Dr. Sara Domenici and has not been submitted for any other degree.

All sources used have been properly acknowledged, and this thesis complies with the academic integrity regulations of Politecnico di Torino.

Date: 1st July,2025

Signed by,
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Abstract

Dye-Sensitized Solar Cells (DSSCs) represent a promising class of low-cost, environmentally friendly photovoltaic devices that offer an alternative to conventional silicon-based solar cells. This study investigates the performance characteristics of DSSCs fabricated using titanium dioxide (TiO_2) nanoparticles, with a focus on evaluating the impact of **electrolyte composition** (aqueous iodide/triiodide vs. xanthan gum-based gel) and **sintering technique** (oven vs. hotplate) on photovoltaic efficiency. Inspired by sustainable solar cell development as demonstrated by *Domenici et al. (2025) and Galliano et al. (2020).* The experimental setup involved multi-stage fabrication of the photoanode, including substrate cleaning, blocking layer deposition, mesoporous TiO_2 layer application, $TiCl_4$ treatment, dye sensitization with D149 dye and counter electrode preparation via platinum sputtering. DSSC assembly was finalized through Surlyn-based sealing and electrolyte injection. Four configurations were studied: Oven + Gel, Oven + Aqueous, Hotplate + Gel, and Hotplate + Aqueous.

Key photovoltaic metrics — **Open Circuit Voltage (Voc)**, **Short Circuit Current Density (Jsc)**, **Fill Factor (FF)**, and **Power Conversion Efficiency (PCE)** — were measured and analysed. Results demonstrated that the **Oven + Gel configuration** yielded better efficiency, attributed to improved interfacial contact, reduced recombination, and superior sintering uniformity. Surface morphology assessments revealed that oven-sintered films exhibited smoother and more porous structures, while hotplate sintering introduced surface defects that impaired performance.

The findings validate the synergistic importance of optimized sintering and gel- based electrolyte systems in enhancing DSSC efficiency. The study not only confirms trends reported in previous literature but also identifies practical limitations and offers insights for scalable DSSC fabrication in real-world applications.

Keywords

Dye-Sensitized Solar Cells (DSSCs); Hydrogel Electrolyte; TiO_2 Nanoparticles; Indoor Photovoltaics; Oven Sintering; Hotplate Ramp Sintering; D149 Dye; Power Conversion Efficiency (PCE); Xanthan Gum; Sustainable Solar Energy.

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Chapter 1: Introduction

1.1 Background of the Study

In the 21st century, rising industrialization, technological progress, and global population growth have significantly increased energy demand. Concurrently, the world is striving to reduce carbon emissions and transition toward cleaner, sustainable energy sources. Solar power, owing to its abundance and renewability, has become a promising solution. Among solar technologies, photovoltaics (PV) remains the cornerstone, converting sunlight directly into electricity. Despite the dominance of silicon-based PV due to its efficiency and maturity, challenges such as high material costs and limited flexibility have spurred investigations into alternative PV types.

Dye-Sensitized Solar Cells (DSSCs) pioneered by O'Regan and Grätzel in 1991 possess a photosynthetic mechanism. Dye molecules transfer electrons to a wide bandgap semiconductor (most typically TiO_2), which in turn transfers the electrons to the anode while a redox electrolyte re-oxidizes the dye and closes the circuit via the counter electrode *(Nazeeruddin et al., 2011).* DSSCs are inexpensive to produce, flexible and transparent, and so find applications such as building-integrated photovoltaics and wearable electronics. Commercialisation is hindered by efficiency and long-term stability issues.

The highest priority among the areas in which DSSCs need to be optimised is electrolyte evaporation and leakage, electron recombination at the semiconductor-electrolyte interface, and manufacturing processes that affect material integrity. These include, among others, gel-based electrolytes (e.g., xanthan gum) and emerging sintering processes (hotplate vs. oven)—these hold promise for optimising device performance and manufacturability *(Galliano et al., 2020; Lowtemperature sintering studies).* In this work, we explore the impact of such alterations on the photovoltaic behaviour of TiO_2 nanoparticle-based DSSCs.

1.2 Need for the Study

Commercialization of DSSCs hinges on solving several technical limitations: cell efficiency, long-term stability, and streamlined fabrication. The conventional liquid electrolyte seeps out and vaporizes with heat, stealing energy and reducing cell lifespan. Substituting it with a gel-based form created from generic biopolymers like xanthan gum provides the device with firm mechanical integrity and retains the juice within, an obvious advantage for flexible panels. Despite that, how quickly ions travel through that gel and if that holds potential for increased efficiency still requires extensive trials *(Galliano et al., 2020).*

The sintering technique for drying and crystallizing the TiO_2 layer determines how well the layer adheres, how permeable its pores are, and, most importantly, how fast electrons can move. A slow bake in a standard oven distributes heat uniformly but is wasteful of both energy and time. A faster spin on a hotplate saves both of those expenses, but it also has the potential to cook a crooked layer that lowers overall performance *(Farhana et al., 2019).*

A comprehensive study combining electrolyte type and sintering method is currently lacking in literature. To bridge this gap, the current research constructs various sets of DSSCs, adjusts both electrolyte formulation and sintering technique, and subsequently contrasts power generation, stability, and overall efficiency.

1.3 Research Problem

Growing interest in DSSCs as a green alternative to traditional photovoltaic technologies is motivated by their prospects for affordable, flexible, and semi-transparent applications. Yet, efforts toward practical implementation are blunted by ongoing challenges related to stability, fabrication reproducibility, and energy conversion efficiency. These limitations are directly linked to the inherent characteristics of the cell's fundamental building blocks: the electrolyte and the photoanode layer *(Galliano et al., 2020).* Liquid electrolytes, as efficient in enabling charge transport, pose disadvantages of volatility, leakage, and low long-term stability. Gel electrolytes, which are meant to overcome these limitations, can change the internal resistance and ionic conductivity of the cell. Accordingly, the selection of the electrolyte is critical in determining the cell's functional properties *(Mohammadpour et al., 2016).*

At the same time, the sintering process used in manufacturing the TiO_2 layer greatly influences its structural stability, porosity, and electronic network. Hot plate sintering provides a faster, localised heating process, but can lead to uneven heat distribution. Oven sintering provides temperature uniformity but consumes more energy and time. Sintering quality and electrolyte behaviour are coupled in intricate and less-researched ways *(Mohammadpour et al., 2016).*

The research problem guiding this thesis is:

"What are the performance implications of using aqueous versus gel-based electrolytes and hot plate versus traditional oven sintering techniques in DSSCs constructed with TiO_2 nanoparticles?

1.4 Objectives of the Study

The overall aim of this research is to synthesize and characterize DSSCs in the field of TiO_2 nanoparticles and identify the combined impact of different electrolytes and sintering treatments. The specific aims are:

1. Develop DSSCs in a standardised process with TiO_2 nanoparticle-based photoanodes.
2. Investigate the influence of aqueous (liquid) vs. viscous (gel) electrolytes on the performance of DSSC.
3. Compare hot plate sintering and oven sintering in terms of efficiency, time usage, and structural integrity of the TiO_2 layer.
4. Measure and compare critical photovoltaic parameters such as Voc (open-circuit voltage), Isc (short-circuit current), FF (fill factor), and PCE (power

conversion efficiency).

5. Identify the best combination of electrolyte and sintering process for optimum DSSC performance.
6. Present proposals for the optimisation of DSSC production for real-world applications using the findings acquired.

1.5 Scope of the Study

This study focuses on a laboratory-scale comparative analysis of DSSCs fabricated using different electrolytes and sintering techniques. The scope is limited to:

- The use of TiO_2 nanoparticles as the semiconductor photoanode material.
- The comparison of liquid (water-based) and gel (Xanthum gum-based) electrolytes.
- The application of two sintering techniques: hot plate and traditional oven.
- The use of D149 organic dye as the sensitizer.
- Fabrication using FTO glass substrates and platinum-coated counter electrodes.
- Testing under controlled laboratory conditions using a calibrated solar simulator (AM 1.5G).

1.6 Methodology Overview

The methodology used in this research combines materials engineering, device fabrication, and electrical testing techniques. The major phases include:

1.6.1 Material Preparation

- FTO glass substrates were cut, cleaned, and prepared for coating.
- TiO_2 nanoparticle paste was used to deposit blocking and mesoporous layers.
- Dye solutions were formulated using D149 and additives like CDCA.

1.6.2 Fabrication Process

- Blocking layers were applied via spin coating.
- Mesoporous layers were applied via screen printing.
- Sintering was conducted using both a hot plate and an oven.
- $TiCl_4$ treatment was applied to enhance layer performance.
- Dye sensitization involved immersion for 4–5 hours.
- Counter electrodes were prepared via platinum sputtering.
- Electrolyte was injected, and cells were sealed using Surlyn foil and glass.

1.6.3 Testing and Analysis

- Cells were inspected with a VeraSol Solar Simulator.
- I–V curves were collected with Orient Instrument software.
- The values such as Voc, Isc, FF, and PCE were calculated.
- Tabular analysis and comparison charts were carried out for evaluation.

1.7 Significance of the Study

The findings from this study will offer valuable insights into optimizing DSSC fabrication processes. Key contributions include:

- Demonstrating the viability of gel electrolytes for leak-free, stable DSSCs.
- Providing empirical data on the comparative effectiveness of hot plate vs. oven sintering.
- Helping small-scale researchers and academic labs adopt faster and more efficient DSSC production methods.
- Enabling informed decisions on material choices and process parameters for DSSC development.

1.8 Organization of the Thesis

- **Chapter 1: Introduction** – Establishes the background, objectives, and methodology of the study.
- **Chapter 2: Literature Review** – Reviews past research related to DSSC materials, fabrication techniques, and performance challenges.
- **Chapter 3: Materials and Methods** – Details the experimental setup, step-by-step procedures, and materials used.
- **Chapter 4: Results and Discussion** – Presents and analyses the performance data and compares the different setups.
- **Chapter 5: Conclusion and Future Work** – Summarizes findings and proposes directions for further research.
- **Chapter 6: References** – Lists all citations used throughout the thesis in APA format.
- **Chapter 7: Appendices** – Contains experimental images, raw data, and supplementary materials.

1.9 Limitations

- Only two types of electrolytes were tested.
- Only two sintering methods were employed.
- Testing was confined to laboratory conditions; real-environment behavior remains unknown.
- Long-term ageing, stability, and degradation were not evaluated.

Performance metrics were limited to short-term I–V testing under simulated sunlight.

CHAPTER

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Chapter 2: Literature Review

2.1 Introduction to Dye-Sensitized Solar Cells

Dye-Sensitized Solar Cells (DSSCs) have emerged as one of the most promising third-generation photovoltaic devices due to their cost-effectiveness, ease of fabrication, and adaptability to low-light environments. The original concept of Grätzel and O' (Regan in the 1990s) DSSCs has a photosensitizer dye that absorbs sunlight to generate electrons; electrons are then transferred onto a nanocrystalline semiconductor, usually titanium dioxide (TiO_2), from which they are transferred to an external load to create an electrical current. Thus, the unique structure of DSSCs is composed of (1) a transparent conducting oxide (TCO) substrate, commonly FTO glass; (2) a porous TiO_2 layer; (3) a dye molecule; (4) a redox electrolyte; and (5) a counter electrode usually coated with platinum. (*Nazeeruddin et al., 2011*).

What differentiates DSSCs from conventional silicon solar cells is their ability to operate effectively under diffuse light conditions and at varied angles of light incidence, which makes them ideal for indoor or low- light applications. The semi-flexible nature of their design also opens opportunities in wearable electronics and building-integrated photovoltaics (*Smestad, 2002*). These advantages have propelled research into optimizing individual components of DSSCs to further enhance their efficiency and longevity.

2.2 Electrolytes in DSSCs

Types of Electrolytes: In a DSSC, the function of the electrolyte is to re-oxidise the oxidised dye by delivering a redox mediator, and to transfer charges between the counter electrode and photoanode (*Sharma et al., 2021*). Conventional DSSCs employ a liquid electrolyte in the form of an organic solvent (e.g. acetonitrile, methoxypropionitrile) with the iodide/triiodide (I^-/I_3^-) redox couple (*Galliano et al., 2020*). This liquid I^-/I_3^- platform has made high initial efficiencies (higher than 11% with Ru-based dyes)(*Sharma et al., 2021*) possible, due to rapid dye regeneration and charge transport. Consequences of liquid electrolytes are, however, volatility and leakage of the solvent, as they are corrosive or light-sensitive, I_3^-, *Galliano et al. (2020).* The unstable solvents will evaporate over time and reduce performance, and I_3^- can penetrate cell components or degrade the dye under extended UV exposure. To counteract the above factors, researchers have used **quasi-solid or gel electrolytes,** in which a polymer matrix or a gel is incorporated to lock the liquid in place. These incorporate the addition of high-boiling ionic liquids or gelling polymers (e.g. PVDF-HFP, PEG, etc.) to create viscous electrolytes (*Galliano et al., 2020*). Such **gel electrolytes** lower the vapour pressure and leakage with ionic conductivity. **Solid-state electrolytes** finally substitute the liquid phase totally with a solid hole-transport material (HTM) or p-type semiconductor. In the case of solid-state DSSCs, a Spiro-OMeTAD organic hole conductor or inorganic p-type semiconductors (such as CuI, CuSCN) are doped into the porous TiO_2 to transport holes(*Sharma et al., 2021*). Solid-state DSSCs are free from liquid solvent problems and have realized appreciable efficiencies (around 5–8% PCE) (*Yeoh and Chan, 2017*), but pore filling may be partial and solid HTMs also suffer from their stability issues (e.g. Spiro- OMeTAD is sensitive to moisture and needs dopants for conductivity). Every type of electrolyte therefore, involves a compromise between performance and stability.

Ionic Transport and Stability: The electrolyte's ionic conductivity and stability strongly influence DSSC operation. In liquid I^-/I_3^- electrolytes, tri-iodide diffusion through the mesoporous TiO_2 network must be fast enough to replenish the dye, but not so fast as to recombine excessively with injected electrons. Small solvent molecules like acetonitrile enable high ionic diffusivity and thus high short-circuit current (Jsc). However, these solvents are highly volatile and can leak from cell seals over time, causing drying of the cell. Moreover, I_3^- is a mild oxidizer that can slowly attack the dye and counter electrode (Pt) or even the sealant. Studies have shown that water contamination in a DSSC can initially increase the open-circuit voltage (by shifting the iodide/triiodide redox potential and blocking some recombination) but at the cost of lowering Jsc due to dye desorption and surface effects (*Bella et al., 2015*). Water molecules adsorb on TiO_2 and can displace dye or coordinate surface sites, leading to reduced photocurrent. Indeed, a hydrophilic dye environment can cause dye molecules to detach; thus, when water-based electrolytes are used, dye molecules are often engineered with hydrophobic ligands or co-adsorbents to protect them. For example, adding hydrophobic alkyl chains or chenodeoxycholic acid (CDCA) as a co- adsorbent on the dye can improve its stability in contact with aqueous or high-polarity electrolytes.

The I^-/I_3^- redox couple also absorbs in the visible region, which can parasitically reduce light harvesting. Alternatives like cobalt (II/III) bipyridyl complexes have been introduced as redox shuttles with lower absorption and higher redox potential (increasing device voltage). *Bella et al. (2016)* demonstrated that cobalt-based electrolytes can yield more stable devices and higher voltages, though they often require good TiO_2 surface passivation to suppress recombination. The trade-off is that these complexes diffuse more slowly than I^-/I_3^- in typical solvents, potentially limiting current. Similarly, ionic liquid electrolytes (which are liquid salts at room temperature) have been used to eliminate volatile solvents. Nonetheless, devices with ionic-liquid-based electrolytes have shown significantly enhanced lifetimes, with stability over thousands of hours reported (*Bella et al., 2016*). To combine the advantages of liquids and solids, gel/quasi-solid **electrolytes** use a porous or polymer host that traps the liquid electrolyte. For example, adding fumed silica or polymers can turn the liquid into a gel that resists leakage. Such gelled electrolytes maintain decent ionic mobility while preventing fluid flow out of the cell, thereby improving long- term stability.

Additives and Polymer Matrices (Xanthan Gum and others): A recent focus has been on bio- **polymer gel electrolytes** that are environmentally benign. Xanthan gum (XG), a natural polysaccharide, is one such gelling agent that has shown promise in fully aqueous DSSCs. *(Galliano et al., 2020)* reported xanthan-gum-based hydrogel electrolytes for "water-only" DSSCs, achieving **remarkable stability** over 1200 hours with performance that improved by 20% after ageing, reaching about 2.7% efficiency. The xanthan hydrogel traps the I^-/I_3^- aqueous solution in a robust 3D network, preventing evaporation while being sufficiently porous to penetrate the TiO_2 film like a liquid. Notably, the devices with XG gel retained high reproducibility and functioned stably even in an air atmosphere without special sealing. Xanthan is just one example – other polymers like **carboxymethyl cellulose (CMC)**, agarose, K-carrageenan, and block copolymers (Pluronic, etc.) have been studied as DSSC electrolyte matrices. Often, these additives aim to address both volatility and allow use of safer solvents (water or ethanol). Many early attempts with biopolymers had lower efficiency (for instance, a κ- carrageenan hydrogel DSSC yielded <0.01% PCE), but incremental improvements (e.g. using Pluronic F77 copolymer, achieving ~2% or silica gel achieving ~1.5%) showed the viability of quasi-solid designs. The XG-based system by Galliano et al. stands out because it combines a **completely renewable solvent (water) and biopolymer** with performance approaching 3% and excellent stability. This indicates that with proper optimization of gel composition (e.g. tuning ion concentrations, polymer content, and dye dipping conditions), green electrolytes can rival traditional ones.

In contrast, **solid-state DSSCs** using HTMs eliminate liquid entirely and thus avoid evaporation and leakage by design. They have achieved up to ~7–8% efficiency using Spiro-OMeTAD as the hole conductor and novel sensitizers *(Benesperi et al., 2018)* provide a comprehensive review of such solid-state DSSCs, noting that while they circumvent liquid-related instability, they face challenges in **pore filling** and charge transport. The solid HTM must infiltrate the 10–20 µm thick Nano porous TiO_2 layer; incomplete filling leaves voids that limit photocurrent. Moreover, many HTMs (Spiro- OMeTAD, for example) require the addition of hygroscopic dopants (Li salts, Co- complexes) to attain sufficient conductivity, which can introduce new instability (dopant diffusion, HTM crystallization over time). Thus, solid-state DSSCs often show faster performance decline in long tests compared to well-sealed liquid cells. Recent advances include copper-complex HTMs and new molecular p-type materials that are more stable, but the efficiencies still lag liquid- electrolyte cells.

In short, electrolytes in DSSCs have evolved from volatile organic liquids to more stable and safe media**. Liquid electrolytes** continue to provide the best efficiency, but have sealing and durability problems. Polymer **gel and quasi-solid electrolytes** provide a promising path towards stability, allowing DSSCs that are leak-free and non-toxic for thousands of hours. Solid-state DSSCs do away with liquids altogether, solving evaporation, but need to be further developed to keep up with the photovoltaic efficiency of liquid-based counterparts.

2.3 TiO_2 Photoanode Materials and Modifications

The photoanode of a DSSC is typically a mesoporous film of titanium dioxide (TiO_2) nanoparticles deposited on a transparent conducting oxide (TCO) glass (usually FTO). This mesoporous TiO_2 layer (also called the working electrode) is the backbone that holds the dye and facilitates electron transport. Key requirements for the TiO_2 photoanode are: high surface area for maximal dye adsorption, good connectivity between particles for electron percolation, and a favourable band alignment with the dye and electrolyte. The classic breakthrough by O'Regan and Grätzel was the use of a **nanocrystalline TiO_2 film with an extremely high roughness factor (~1000)**, enabling nearly monolayer dye coverage across an enormous internal area and efficient light harvesting. This innovation increased the photocurrent by orders of magnitude compared to flat electrodes, as a single dye monolayer on a flat surface absorbs only ~1% of light. The mesoporous film is usually 10–15 μm thick and composed of ~20 nm anatase TiO_2 particles sintered together. When sensitized with suitable dye molecules (e.g. Ru-based N3 or N719), such films can achieve incident photon- to-current conversion efficiencies (IPCE) above 80% in the dye's absorption range.

2.3.1 Mesoporous Structure and Electron Transport:

In a mesoporous TiO_2 anode, electrons travel through the interconnected nanoparticles by a random-walk (diffusive) transport mechanism *(Mohammadpour et al., 2019)*. Each electron undergoes multiple trapping and de-trapping events at surface states or grain boundaries as it percolates to the TCO contact. Despite this trap-limited diffusion, the electrons can still be collected efficiently if their lifetime (before recombination) is much longer than the transit time. Optimizing particle necking (through sintering) and crystallinity helps reduce trap density and increase electron mobility. A well-sintered TiO_2 network shows improved electron transport compared to a poorly connected one. For instance, *Mohammadpour et al. (2015)* found that using an **optimized annealing profile** for TiO_2 nanotube membranes (rapid thermal annealing at 500 °C) led to markedly lower trap-state density and improved electron i level and Voc *(Selopal et al., 2014)*. It is worth noting that while a blocking layer typically does not affect the dye uptake (since it is beneath the mesoporous layer), it must be optimized in thickness – too thick and it adds series resistance, too thin and pinholes reduce its effectiveness.

2.3.2 $TiCl_4$ Post-Treatment:

A common procedure to improve TiO_2 photoanodes is **titanium tetrachloride ($TiCl_4$) treatment** of the nanoparticle film. In this treatment, the sintered TiO_2 film is soaked in a $TiCl_4$ aqueous solution (typically 40 mM) and then annealed again. $TiCl_4$ hydrolyzes and deposits an ultrathin TiO_2 layer (or tiny TiO_2 particles) onto the existing nanostructure. This has multiple benefits: it "fills in" necks and voids between particles, improving electrical connectivity, and it increases surface area by adding new TiO_2 on the particle surfaces, thus allowing more dye adsorption. Moreover, the freshly deposited TiO_2 can passivate surface traps on the nanoparticles. The result of $TiCl_4$ post-treatment is typically an increase in Jsc (due to higher dye loading and better connectivity) and often a slight increase in Voc (due to reduced surface recombination). *(Hagfeldt et al)* And others have routinely used $TiCl_4$ treatment to boost efficiencies in their cells. For instance, an efficiency improvement on the order of 10–20% relative was observed in many cases after $TiCl_4$ post-treatment, which became a standard step in DSSC fabrication.

2.3.3 Surface and Bulk Additives: Aside from $TiCl_4$, researchers have explored additives to the TiO_2 paste or film to enhance its properties. Recent work by *Fagiolari et al. (2020)* systematically studied **molecular and polymeric additives** introduced into a commercial TiO_2 paste to improve performance in aqueous DSSCs *Fagiolari et al. (2020)*. They tried small molecules like α-terpineol (a solvent/cosolvent) and propylene carbonate, as well as polymers like polyethene oxide (PEO), polyethene glycol (PEG), carboxymethyl cellulose, and xanthan gum mixed into the TiO_2 paste before film deposition *Fagiolari et al. (2020)*. Among all, PEG was found to yield the best improvement: the PEG-modified TiO_2 films showed **18% higher Jsc and 48% higher overall efficiency** compared to the unmodified paste. The PEG likely acts as a pore-forming agent and binder that modifies the film's porosity and thickness; X-ray analysis indicated changes in the anatase crystal orientation with PEG and altered film morphology *Fagiolari et al. (2020)*. In practice, the PEG-additive paste produced a thicker but crack-free mesoporous layer with enhanced dye uptake and light scattering, thus boosting current. Other additives like CMC and XG in that study did not outperform PEG, but they highlight the approach of tailoring the **paste formulation**. By adjusting the paste rheology and drying behaviour, one can influence the particle packing and film integrity (avoiding cracks and improving contact). This is especially relevant for **aqueous DSSCs**, where the standard paste (optimised for high-temp sintering and organic solvent compatibility) may not be ideal when water is the electrolyte. Indeed, alternative binder systems or surfactants can make the TiO_2 film more hydrophilic, aiding penetration of water-based electrolytes.

2.3.4 Light Scattering Layers: Another photoanode refinement is the use of a **bilayer structure**: a transparent nanocrystalline TiO_2 underlayer (for dye adsorption) topped with a **light-scattering overlayer** of larger oxide particles (100–400 nm). The larger particles (or aggregates) scatter incident light back into the dye-loaded layer, increasing the optical path length and improving photon absorption. This technique can significantly improve Jsc without sacrificing transparency diffusion length, boosting cell efficiency by ~50% (to 9.8%) *(Mohammadpour et al., 2019).* In contrast, slower annealing produced nanotube walls with more cracks and defects, which increased trapping and recombination. This highlights that the **microstructure of TiO_2 (crystal phase, grain connectivity, defects)** directly impacts DSSC performance.

2.3.5 Blocking Layers: One crucial modification to the photoanode is adding an **electron blocking layer** (also called an underlayer or compact layer) between the TCO glass and the mesoporous TiO_2 film. This is typically a thin (50–100 nm) compact TiO_2 layer deposited by methods like spray pyrolysis or spin coating before the mesoporous layer *(Wu et al., 2023)*. The blocking layer covers the conductive glass surface, preventing the electrolyte from directly contacting the TCO. This avoids the unintended back- reaction where electrons in the TCO (or at the TiO_2/TCO interface) recombine with the redox species in the electrolyte *(Wu et al., 2023)*. By eliminating these shunt pathways, a blocking layer raises the open-circuit voltage and fill factor of the cell. For example, a study reported that adding a compact TiO_2 layer improved efficiency notably by suppressing dark current at the interface. The blocking layer should be pinhole-free and intimately cover the TCO; materials like a thin dense TiO_2 or even insulating oxides (ZnO, SnO_2, or Nb_2O_5) have been used for this purpose *(Ozel, Atilgan, and Yildiz, 2024)*. Additionally, some works dope the blocking layer (e.g. with zinc or magnesium) to tailor its band edge. The net effect is a higher electron density in TiO_2 (since fewer electrons are lost to the electrolyte) and thus a higher Fermi excessively. For example, adding a 4–5 µm-thick second layer of 400 nm anatase particles on a 10 µm nanoparticle film has yielded 20–30% higher photocurrents in some DSSCs. Commercial pastes like "WER2-O" (transparent) and "WER2-O Reflector" (scattering, larger particles) from Dyesol (Greatcell) are often used in tandem. While our focus is on TiO_2 itself, it should be noted that alternative metal oxides have also been researched – ZnO nanostructures, SnO_2, or composites thereof – but TiO_2 remains the benchmark due to its favourable band alignment and proven stability in the iodide electrolyte. ZnO can dissolve in acidic dyes and has seen limited success, and SnO_2 has higher electron mobility but lower dye affinity and more severe recombination (due to a lower CB edge vs I_3^- redox potential) That said, composite photoanodes (e.g. a core of SnO_2 coated with TiO_2 shell) have been explored to exploit the high mobility of SnO_2 while maintaining a TiO_2-like interface for the dye and electrolyte.

In summary, the TiO_2 photoanode has seen continual improvement since the original Grätzel cell:

- A **mesoporous nanoparticle structure** is essential for high dye loading and remains the standard *Sharma, K., Sharma, V., and Sharma, S. S. (2021)*.
- **Blocking layers** at the TCO/TiO_2 interface suppress recombination and are widely employed for higher voltages *Wu et al. (2023)* .

- **Post-treatments like $TiCl_4$** improve particle connectivity and surface chemistry, boosting current output *Selopal et al. (2014)*

- **Nanostructuring and additives:** including scattering particle layers, and paste additives (PEG, etc.), have further enhanced light utilization and film quality *Fagiolari et al. (2020).*

- **One-dimensional structures (nanotubes, nanorods):** These offer direct electron pathways and have shown some success (e.g. TiO_2 nanotube membranes yielding ~9–10% efficiency when optimally annealed)*(Mohammadpour et al. 2015).* However, they often have less internal surface area than nanoparticle films of the same thickness, so a balance must be struck.

All these innovations on the photoanode side aim to increase photocurrent and voltage while mitigating recombination. A well-engineered photoanode, combined with an appropriate electrolyte and dye, directly translates to higher efficiency. For instance, water-based DSSCs that historically lagged in performance have recently closed the gap by using improved photoanode formulations (PEG-modified TiO_2) alongside novel electrolytes *Fagiolari et al. (2020)*. Going forward, research continues into doping TiO_2 with trace cations or passivating its surface with ultrathin coatings (Al_2O_3, MgO) to further reduce losses *Sharma, K., Sharma, V., and Sharma, S. S. (2021)*. The photoanode remains a fertile ground for innovation in DSSCs, as incremental gains here affect multiple performance parameters (Jsc, Voc, stability).

2.4 Fabrication and Sintering Techniques for TiO_2 Films

Fabrication of the TiO_2 photoanode typically involves depositing a paste of TiO_2 nanoparticles onto the FTO glass by screen-printing or doctor-blading, followed by **sintering (annealing) at high temperature** to form a mechanically robust, electrically connected network. The sintering step is critical: it burns off organic binders in the paste and causes adjacent TiO_2 particles to neck together at contact points, greatly improving electron percolation. The conventional sintering protocol is an **oven bake around 450 °C for 30 minutes** (often with a gradual ramp-up and cooldown) in air. This reliably yields crystalline anatase TiO_2 films with good adherence to the substrate and no residual organics. An alternative commonly used in labs is **hotplate sintering**, where the coated substrate is placed on a pre-heated hotplate (e.g. 450 °C) for a similar duration to achieve the nanocrystal annealing. Both oven and hotplate methods can produce functional photoanodes, and in fact many DSSC fabrication guides note that either can be used to sinter the films. The fundamental requirement is that the TiO_2 reaches the requisite temperature long enough to crystallize and fuse. In practice, there are some differences:

- An **oven** provides uniform radiant/convection heating around the sample. This uniformity can ensure even sintering through the film thickness and across the substrate. Ovens also allow controlled temperature ramp profiles (slow heating and cooling), which can minimize thermal shock or stress in the glass and film. Gradual cooling is beneficial to prevent micro-cracks. For example, a typical profile might ramp at ~10 °C/min to 450 °C, hold 30 min, then cool at 5 °C/min. This yields reproducible results with fully burned-out binders*(Mohammadpour et al. 2015)* .

- A **hotplate** heats primarily from the bottom (conductively through the glass). Initially, the glass side in contact heats faster than the top of the film, potentially creating a temperature gradient. However, for thin glass slides, heat distributes relatively quickly and the entire film can reach the target temperature given sufficient time. Hotplates often heat more rapidly (since the sample is in direct contact with a high-temp surface without the air heat-up lag). This can lead to a shorter ramp-up (or an effectively "shock" heating if placed on an already hot plate). Rapid heating on a hotplate may actually benefit neck formation in some cases – analogous to the rapid thermal annealing mentioned in the nanotube study, which yielded fewer defects*(Mohammadpour et al. 2015)*. On the other hand, if the top of the film lags in temperature, binder removal at the film surface might be less complete unless the plate is covered or sufficient time is given. In practice, researchers have reported that **hotplate-sintered TiO_2 films can perform on par with oven-sintered films**, provided the temperature and time are well controlled. The DSSC assembly procedure by Deliot *et al.* explicitly notes sintering "in an oven or hotplate at 450 °C" as equivalent steps. Thus, for standard electrodes on glass, either method can yield ~11% efficient cells (with N719 dye) when optimized.

2.4.1 Comparison and Effect on Crystallization: Both techniques realise the conversion of the screen-printed TiO_2 paste (consisting of anatase particles along with organic binders/terpineol) into a porous sintered film. The most important quality factors after sintering include: phase purity (rutile content vs anatase content), extent of particle necking, film adhesion, and crack absence. Slow ramp oven sintering tends to retain the anatase phase to ~500 °C; after that, some of the anatase can begin to convert to rutile. Hotplate sintering, if carried out at the same temperature, should also retain anatase (since the top temperature is the same). But one factor to consider is that the temperature gradient on a hotplate may not be as even: the film side closer to the plate may see somewhat higher effective temperatures or dissimilar cooling regimes than the top, potentially affecting grain growth. Regardless, no noticeable rutile formation occurs at 450 °C on either platform under normal use (the anatase phase is still dominant). What has been found is that if the sintering temperature is decreased (in order to fit plastic substrates, for example), the particle necking is incomplete and efficiency of cells decreases significantly *Lee et al. (2018)*.

2.4.2 Microstructure Differences: Some subtle differences between oven and hotplate sintering have been reported anecdotally. Oven sintering in ambient air allows the entire film, including its top surface, to be directly exposed to heat and oxygen. This ensures all binder residues burn out from throughout the film thickness. Hotplate sintering might leave the very top surface of the TiO_2 film slightly less processed if it is not exposed to convective heat or if combustion products do not escape as efficiently. In practice, users mitigate this by placing a cover over the cell or using a two-step process (e.g. first a low-temperature bake on the hotplate to dry and burn organics, then a higher temperature final bake). The result is typically comparable film quality. Indeed, many labs favor hotplates for convenience (faster turnaround and no need for a furnace), and report similar cell performance to oven-sintered electrodes. It must be noted specialized sintering techniques developed to bypass high temperatures altogether, particularly for plastic or flexible substrates that cannot handle 450 °C. One of these is chemical or low-temperature sintering with precursors that can "bind" particles at <150 °C. For instance, the addition of trace amounts of TiO_2 sol or titanium (IV) chloride to the paste, or UV light and chemical curing (as in the hot UV method) can partially sinter at low temperature. Another state-of-the-art technique is plasma or microwave-assisted sintering. Zen et al. (2012) demonstrated a dielectric barrier discharge (DBD) plasma treatment that, when deposited following a 250–300 °C low-temperature bake, efficiently removed residual organics and caused TiO_2 necking such that the cell efficiency was on a par with a 500°C-sintered control *Lee et al. (2018)*. In their work, a TiO_2 photoelectrode sintered at only 150°C (using a special binder-free paste) improved by 1.4× in efficiency after the DBD plasma treatment. This underscores the importance of binder removal and particle connection: even at half the typical temperature, if alternative energy (plasma UV, etc.) can achieve those goals, the cell can perform well.

2.4.3 Oven vs. Hotplate – Performance Impact: While preparing standard glass-based DSSCs, researchers have found that there is negligible performance difference if the film is well sintered. A well-sintered TiO_2 film has the same dye loading and electron transport properties irrespective of whether it is heated using oven or hotplate. The variations can become apparent if the sintering is inadequate: e.g., a hotplate that does not achieve actual 450 °C evenly may leave some residue of organic material, resulting in slightly lower photocurrents or increased recombination. Conversely, a controlled atmosphere of an oven may produce a cleaner burn. But these are generally minor if anything – one investigation found that tiny test cells sintered on a hotplate at 450 °C performed just as well as those that were fired in a furnace. Far more significant is the actual temperature reached and time profile. For example, sintering at 400 °C rather than 450 °C can produce incomplete organic removal (with a brownish color to the film and poorer performance) *Chou et al. (2008)*. On the other hand, moving up to 500+ °C can begin to weld particles too much (losing surface area) or creating minor warpage of glass. Therefore, 450 °C (±20 °C) for ~30 min is an empirical optimum used commonly.

Sintering crystallization behavior entails the growth of crystallites of anatase and annealing of the surface hydroxyls of TiO_2. The burnout of the binder is approximately at 300 °C (for organic elements such as ethyl cellulose), and crystal growth proceeds up to ~450 °C. Sudden heating (as on a pre-heated hotplate) can lead to more sudden burnout – unless controlled, this will occasionally create micro-cracks due to extensive gas evolution. Employing a slower ramp or an initial low-temperature dry step can eliminate that. In fact, certain protocols first dry the films at 80–150 °C and then ramp to 450 °C. The effect on DSSC efficiency of these sintering subtleties usually manifests itself in the fill factor and photocurrent: well-sintered film indicates higher electron conductivity (therefore higher fill factor) and allows greater dye loading (therefore higher Jsc) than ill-sintered one.

In one example, substituting a conventional furnace with a **programmable hotplate** (with controlled ramp rates) showed no loss in efficiency – modern hotplates can be set to heat gradually and hold, essentially performing like a mini furnace. Thus, in research and production, hotplate firing is a viable alternative, sometimes preferred for rapid prototyping or continuous manufacturing (roll-to-roll processes use heating plates or lamps rather than batch ovens).

2.4.4 Summary of Sintering Techniques: Traditional sintering (oven or hotplate) remains the gold standard for glass-based DSSCs owing to highest quality films it offers. New sintering techniques (chemical curing, laser sintering, photonic curing, plasma sintering) are research areas to enable low-temperature processing for flexible DSSCs and reduced energy expenses. Laser sintering, for instance, has been used to sinter Pt counter-electrodes and even TiO_2 films locally with high-energy pulses without substrate heating. Photonic curing with Xenon flash lamps rapidly heats the film to high temperature for a few milliseconds on plastic substrates with partial sintering. While these methods enable the production of functional devices, their efficiency is yet to be as high as traditional sintering; for instance, chemically sintered TiO_2 electrodes can achieve 4–6% efficiency compared to 7–9% for fully sintered ones. The difference is narrowing as processes become more mature.In general, oven vs. hotplate sintering is more a matter of giving the TiO_2 the right thermal treatment, not a matter of which is preferable. Both can produce high-quality crystallization and device performance if done correctly. Equipment and availability of substrate will determine the choice. What is certain from the literature is that a fully interconnected anatase network is required for optimum DSSC performance. Any sintering technique that can provide this – 450 °C furnace, hotplate, or some novel Low-T trick with auxiliary energy – will allow high-efficiency devices. Developing fabrication work in the future is aimed at reducing the thermal budget (essential for scale-up and for plastics) without sacrificing the wonderful electronic properties of the photo anode. Plasma-assisted sintering is one such method that can show even films made at 150 °C can be made to perform if suitably treated, a promising indication for flexible DSSC technology.

2.5 Dye Sensitizers: Alternatives and Innovations

The dye sensitizer is the molecule that captures the light and triggers the photoelectric conversion process. Ruthenium dyes have been the typical focus of DSSC studies because they have wide absorption and high electron injection efficiency. Nevertheless, their high price, complicated synthesis, and environmental issues have prompted the pursuit of alternative, eco-friendly options.

Organic dyes, especially donor–π–acceptor (D–π–A) designed dyes, have become increasingly popular due to their tunable absorption, high molar extinction coefficients, and biocompatibility with TiO_2. *Lee et al. (2020)* tested a series of D–π–A organic dyes and showed that they were able to display competitive efficiencies under the best conditions.

Natural pigments of fruit, vegetables, and flowers with high contents of anthocyanin, flavonoids, and carotenoids are a cost-effective, biodegradable substitute for DSSCs. *Jalali et al. (2025)* showed that DSSCs made from anthocyanin exhibited efficiencies of 6–9% under normal light conditions with the added benefits of local availability and non-toxicity. While natural pigments could be prone to degradation under light and heat, encapsulation and molecular engineering methods are being explored to extend their lifespan.

2.5.1 Device Architecture and Design Innovations

In addition to material design, structural advancements have also been crucial for enhancing DSSC efficiency. Multilayer photo anodes, tandem cell design, and light-trapping structures are under extensive research. *Zhang et al. (2022)* discussed the use of light-scattering nanoparticles and anti-reflective coatings for enhanced light absorption without cell thickness increase.

Flexible DSSCs made on polymer substrates like PET or PEN enable wearables and curved surface devices. The possibility of using transparent conducting oxides other than FTO, i.e., indium tin oxide (ITO), also provides more design freedom. Incorporation of plasmatic nanoparticles like gold or silver in the TiO_2 layer was also shown to increase light absorption because of localized surface Plasmon resonance *(Rahman et al., 2014).*

Researchers have also been working on integrating DSSCs into facades, window structures, and fabrics, taking their application scope from laboratory models to practical applications.

2.6 Performance Benchmarks in Literature

Performance metrics such as open-circuit voltage (Voc), short-circuit current density (Jsc), fill factor (FF), and overall PCE are essential in comparing DSSC technologies. The following benchmark results illustrate the evolution of DSSC performance:

- **Nazeeruddin et al. (2011)** reported a Voc of 0.72 V, Jsc of 15.1 mA/cm^2, FF of 72%, and PCE of 11.2% using Ruthenium-based dye and liquid electrolyte.

- **Jalali et al. (2025)** achieved 9.8% PCE with a natural dye and gel electrolyte, emphasizing the viability of eco-friendly DSSCs.

- **Mehmood et al. (2014)** demonstrated 10.7% efficiency using bilayer TiO_2 and ionic liquid gel electrolyte systems.

2.7 Relevance to the Present Study

The current study compares DSSCs prepared by TiO_2 nanoparticles with aqueous or xanthan gum-based gel electrolytes and sintered by hotplate and oven techniques. Literature surveyed influences some of the following in this thesis:

- Gel electrolytes provide improved thermal and structural stability *(Jalali et al., 2025)*.
- Sintering affects film morphology and must be optimized for energy-efficient fabrication *(Hariharan et al., 2019)*.
- The $TiCl_4$ post-treatment considerably increases electron mobility and dye adsorption *(Domenici, 2025)*.
- Organic dyes usage presents an ecologically friendly and cost-effective option compared to metal complexes *(Lee et al., 2020)*.

2.8 Summary of Reviewed Literature

Table 1: Summary of Reviewed Literature

Authors	Year	Focus Area	Key Findings	Relevance to Study
Nazeeruddin et al.	2011	DSSC architecture	Demonstrated high PCE with Ruthenium dyes	Methodology baseline
Mehmood et al.	2014	Electrolytes, TiO_2 design	Highlighted ionic gel electrolytes and bilayer structures	Supports gel electrolyte integration
Jalali et al.	2025	Natural dyes	Validated efficiency of anthocyanins in DSSCs	Sustainable dye inspiration
Smestad	2002	Solar education	Explained DSSC concepts and basic mechanisms	Theoretical foundation
Domenici	2025	Gel electrolyte optimization	Studied xanthan gum-based gel stability	Material selection support
Hariharan et al.	2019	Sintering technique comparison	Compared oven and hotplate sintering effects	Supports sintering method choices
Lee et al.	2020	Organic dyes	Reviewed D–π–A structures for efficiency gains	Organic dye benchmarking
Zhang et al.	2022	Device architecture	Suggested multilayer and light-trapping designs	Structural improvement strategies
Ossila Ltd.	N.D.	I–V testing protocols	Standardized methods for DSSC characterization	Experimental setup and data logging support

2.9 Summary of Findings and Future Directions

Summary of Literature Findings: Past decades of research on dye-sensitized solar cells realized milestone achievements: Ru-dyes that were optimized, volatile solvent electrolytes, and nanostructured TiO_2 were used to realize over 11–12% conversion efficiency under ordinary sun. Early investigations made simple design principles – high dye loading of mesoporous photoanodes, effective dye regeneration by iodide-based redox electrolytes, and efficient triiodide reduction by platinum catalysts. By contrast, stability and scalability issues have been the focus. The review of literature shows a definite trend to overcome such drawbacks: Electrolytes have moved towards more stable and safer forms (ionic liquids, gels, solid HTMs); photoanodes have been met with blocking layers, new morphologies, and additives for improved charge collection and compatibility with new electrolytes; and processing methods have been improved for better film quality and possible low-temperature processing. These innovations are typically related – for example, innovations in water-based electrolytes led innovations in photoanodes (such as PEG-derived paste formulations) to ensure optimal performance in that new environment. Similarly, the development of solid-state DSSCs demanded highly uniformed photoanodes and pore filling techniques.

2.9.1 Gaps and Ongoing Challenges: Despite the substantial advancements made in the field of dye-sensitized solar cells (DSSCs), certain critical gaps in the literature persist—particularly concerning the interplay between materials, processing techniques, and device performance. These gaps present both challenges and opportunities for further innovation, and this thesis aims to contribute meaningful insight by addressing several of them systematically.

2.9.2 Efficiency vs. Stability Trade-off: The highest efficiency DSSCs still rely on volatile, anhydrous electrolytes and ruthenium dyes that, while efficient, pose environmental and longevity issues *Galliano et al. (2020)*. More eco-friendly systems (e.g. fully aqueous electrolyte with organic dyes) have improved in stability but their efficiencies are only around half of the record values *Galliano et al. (2020)*. Bridging this gap is a priority. Future work is likely to focus on new redox mediators that combine fast kinetics with low volatility (recent copper complexes and ferrocene derivatives are examples) and on **dye molecules tailored for these mediators and solvent environments**. The field is moving toward cobalt- and copper-based redox couples for higher voltage, but these require co-optimization of the dye and anode to prevent recombination. Achieving iodine-level stability with cobalt/copper systems remains a challenge (cobalt electrolytes can suffer from solvent loss or complex degradation over time).

- **Material Sustainability:** The use of platinum counter electrodes and rare ruthenium in sensitizers is a cost and sustainability concern. Alternatives are being developed: carbon-based catalysts (graphene, carbon cloth) and transition metal compounds for the counter electrode have shown promise *Galliano et al. (2020)*. Likewise, metal-free organic dyes or natural dyes are desirable to eliminate Ru, and some have achieved respectable efficiencies (7–9% in champion cells). However, many organic dyes still suffer faster degradation, especially in sunlight and heat. Research into **stabilizing organic dyes** (through molecular engineering or co-adsorbents) is needed to approach the 20-year lifetimes of commercial silicon panels. Recent work on donor–π–acceptor dyes with bulky hydrophobic blocks is one approach to improve stability in DSSCs *Sharma, K., Sharma, V., and Sharma, S. S. (2021)*.
- **Photoanode Enhancements:** While mesoporous TiO_2 is well-optimized, new structures like hierarchically structured films (combining nanoporous and light- scattering features in one layer) or 1D/2D hybrid architectures could further improve performance. For example, embedding a fraction of nanowires or nanosheets within a nanoparticle film might provide direct paths to the conductor, boosting electron transport without sacrificing area. Additionally, **surface passivation** of TiO_2 is a continuing area: ultrathin coatings (a few nanometers of Al_2O_3 or MgO) can passivate surface traps and slow recombination, as shown in some studies, but applying these without blocking dye uptake is tricky. Atomic layer deposition (ALD) may be a future tool to precisely coat internal surfaces. The review also points to the need for **better understanding of electron dynamics** in new anode structures – advanced spectroscopy and modeling can help elucidate how additives like PEG or new phases affect electron lifetime and transport.
- **Low-Temperature Processing:** For DSSCs to be commercially viable in flexible electronics or BIPV on plastics, the 450 °C sintering step must be eliminated. The literature shows promising alternatives (chemical sintering with acids, photonic curing, plasma treatment) that achieve decent efficiency at <150 °C *Lee et al. (2018)*. However, none yet fully match the performance of High-T sintering.

Future research will likely refine these methods – for instance, using **nanoparticle dispersions that sinter in situ** or nanoparticle sols that can be UV-cured. If one can create a TiO_2 network at low temperature that is as conductive as the High-T one, it would remove one of the last manufacturing barriers for DSSCs (allowing roll-to-roll printing on plastics). Zen's plasma approach and others hint that this is feasible *Lee et al. (2018)*, but scaling it up and ensuring uniformity on large areas is a challenge.

- **Long-Term Stability and Sealing:** Many recent studies extend stability tests to thousands of hours under continuous light and elevated temperatures (60 °C or higher*)* *Galliano et al. (2020)*. A gap remains in understanding and preventing all degradation modes: electrolyte dry-out, dye desorption, photo-oxidation of dyes, and TCO corrosion. Encapsulation techniques (like glass frit sealing or UV-curable epoxy seals) are being improved to hermetically seal DSSCs *Capitão et al. (2023)*. Novel sealing materials that can flex (for flexible cells) while keeping moisture out are needed. Also, **solid-state DSSCs** face unique stability issues, such as HTM oxidation or electrode delamination, which require different solutions (e.g. UV filters to protect the HTM, better adherence layers, etc.). Continued aging studies and accelerated stress tests are important to guide these improvements *Sharma, K., Sharma, V., and Sharma, S. S. (2021)*.
- **Overlooked Role of Sintering Technique on Performance:** Sintering directly influences the porosity, adhesion, and surface properties of the TiO_2 layer but few studies comparatively evaluate hotplate sintering and oven sintering of DSSCs. Studies available usually follow a fixed approach to sintering instead of considering it as a variable to be optimized. By comparing the results obtained from hotplate and oven sintering methods using similar materials and structures, this paper provides insight into their strengths and weaknesses.

- **Broken Research Throughout Dye, Electrolyte, and Process Parameters:** Many studies center on optimizing either the dye, the electrolyte, or the sintering process—albeit not often combined in a way to look at synergistic benefits of optimizing all three. This project is more integrated in its methodology, varying more than one factor (electrolyte and sintering) on a single substrate of TiO_2 and dye choice, with the aim of adding to a broader picture of how to optimize DSSCs.

CHAPTER

03

Chapter 3: Materials and Methods

3.1 Introduction

This chapter describes the sequential processes and materials involved in the preparation of dye-sensitized solar cells (DSSCs). The experiment was started from well- established scientific guidelines and then modified through repeated experimentation to achieve the target purpose of comparing different electrolytes (liquid vs. gel) and sintering methods (hotplate vs. oven). Each process step of fabrication, ranging from substrate cleaning to cell assembly, was carried out under a controlled laboratory environment to ensure reproducibility and consistency. The processes are all strongly dictated by pioneering work that scientists like Federico Bella and colleagues have done, with their research on sustainable DSSC electrolytes and sintering optimization having set much of the modern process of conducting this research *(Bella et al. 2020; Domenici et al. 2025)*.

DSSC structure includes the following major components:

1. Working Electrode: A conductive and transparent substrate like FTO glass, which will be used as the semiconductor coating base.
2. Semiconductor Layer: Nanostructured TiO_2 film that allows the transportation of the electrons photo-generated.
3. Sensitizer (Dye): A light-sensitive dye that absorbs light and injects the electrons into the TiO_2 layer.
4. Electrolyte: An iodide/triiodide redox couple solution, commonly used to regenerate the oxidized dye and to carry charge.
5. Counter Electrode: A conductive surface that is plated with a catalyst like platinum to reduce the redox couple.

3.2 Schematic Diagram and Working Principle of DSSC

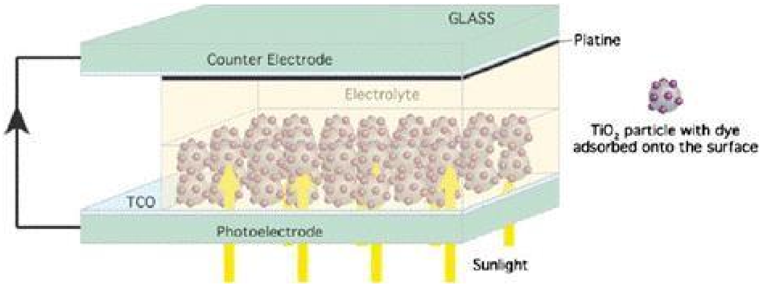


Figure 1: Schematic representation of DSSC working principle. (Nazeeruddin et al., 2011)

The operational mechanism of DSSCs entails multiple functional layers. A mesoporous TiO_2 thin layer coated onto a transparent conducting substrate (typically FTO glass) is sensitized with an absorbent dye that injects electrons into the TiO_2 conduction band upon light absorption. The electrons move in the external circuit to the counter electrode. Oxidized dye is regenerated by the electrolyte (commonly I^-/I_3^- redox couple), which is regenerated at the counter electrode. This cycle produces sustained photocurrent under illumination *(Yeoh and Chan, 2017; Sharma et al., 2021)*.

This diagram not only makes it possible to imagine the working structure of a DSSC but also provides a reference when comprehending the function of every component during fabrication and performance testing.

3.3 Conductive Substrate Preparation (FTO Glass)

Cleaning of the fluorine-doped tin oxide (FTO) glass substrate is one of the crucial procedures in obtaining high-performance fabrication of DSSCs. The substrate is utilized as the initial material for depositing subsequent layers and must be electrically highly conductive, optically transparent, and surface-clean. Particulate contamination, organic contaminations, or surface heterogeneity can cause destruction of the adhesion of the TiO_2 layer and influence charge transfer reactions. Commercial FTO glass plates with a sheet resistance of approximately 15 Ω/sq were used in this research. The sheets were cut into neat rectangular pieces of dimensions 2 cm × 1.5 cm in a precision glass cutter. Gloves and tweezers were worn to work with each segment to avoid contamination. The conductive side of the glass was ascertained by employing a multimeter by measurement of surface resistance. Properly identified conductive sides alone were coated.

A multi-stage ultrasonic cleaning regimen was adopted to ensure a clean surface:

1. **Step1: Soap Sonication**

   The substrates were submerged in a lab-grade detergent solution and sonicated within an ultrasonic bath for 15 minutes. This removed greasy residues, dust particles, and surface oils.

2. **Step 2: Deionized Water Rins**

   Following sonication of the soap, all substrates were washed extensively with high-purity deionized (DI) water to remove soap residues and ionic impurities.

3. **Step 3: Solvent Sonication**

   Substrates were then sonicated in isopropyl alcohol (IPA) for 15 minutes and then acetone for another 15 minutes. These solvents possess the capacity to dissolve organic impurities and enhance the glass surface wettability.

Following decontamination, substrates were dried with a nitrogen gas blower and sealed in clean containers for storage. This stringent cleaning procedure afforded the optimal surface energy necessary for uniform TiO_2 layer deposition and good adhesion.

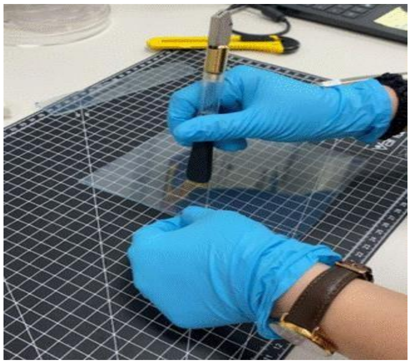


Figure 2: *Cutting the FTO substrate*

Figure 3: Ultrasonic bath used for cleaning
(Source: Electrochemistry Lab – Polito)

3.4 Blocking Layer Deposition

The thin TiO_2 blocking layer effectively inhibits back-transfer of electrons from the FTO to the redox couple in the electrolyte. The major function of the blocking layer is to serve as an energy barrier to annihilate interfacial recombination, resulting in enhanced open circuit voltage (Voc) and efficiency of the DSSC.

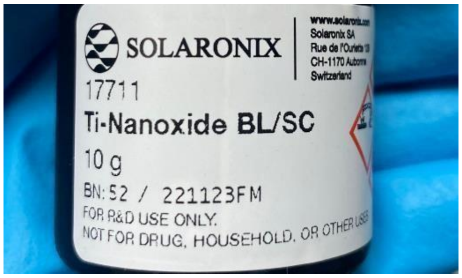


***Figure 4**: Ti-Nanoxide as a Blocking layer (source: electrochemistry lab- Polito)*

A commercially prepared TiO_2 blocking solution was utilized for this step (Figure 4). The deposition was carried out using a **spin-coating technique**, which offers high control over film thickness and uniformity.

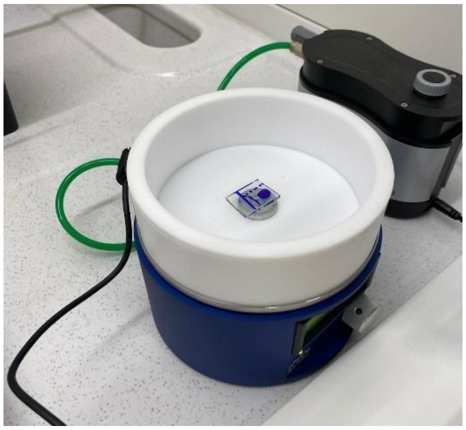


***Figure 5**: Spin-coater for depositing the blocking layer (source: electrochemistry lab- Polito)*

Spin-Coating Procedure:

- 75 µL of the TiO_2 blocking solution was pipetted onto the conductive side of the cleaned FTO substrate.

- The substrate was spin-coated at 5000 revolutions per minute (rpm) for a duration of 30 seconds using a programmable spin coater.

- Post-spin, the substrates were left to dry under ambient conditions for 10–15 minutes.

To convert the deposited amorphous layer into a crystalline phase and to promote strong adhesion, a sintering process was applied. Two sintering approaches were explored:

- **Hot Plate Sintering:**
  The dried substrates were placed on a digitally controlled hot plate. A PR5 programmable temperature controller was used to gradually ramp the temperature to 500°C. The heating rate was carefully controlled to avoid thermal shock or warping.

- **Oven Sintering:**
  Alternatively, samples were placed in a pre-heated muffle furnace at 500°C for 30 minutes. Post-sintering, the substrates were allowed to cool down gradually inside the oven to prevent cracking due to thermal stress.

3.5 TiO_2 Screen Printing and Sintering

The mesoporous TiO_2 film is the focal point of the DSSC, being the scaffold for dye adsorption and primary electron conduction pathway. The film must possess a high surface area for dye loading and facilitate efficient electron mobility by interconnected nanostructures.

For coating of this layer, commercial TiO_2 nanoparticle paste (typically containing anatase phase TiO_2 particles of average diameter 20 nm) was used. The paste was applied using a screen-printing technique, where there is controlled thickness and patterning.

Procedure:

- A stainless-steel mesh and shadow mask (having a 0.25 cm^2 active area) were used to have uniform thickness and reproducibility for all the samples.
- A narrow strip of TiO_2 paste was deposited at the border of the screen, and spread evenly onto the substrate surface by using a rubber squeegee.
- Several passes were made to distribute the film evenly in thickness.

- The printed films were air-dried for 15 minutes following deposition to enable evaporation of the solvents in the paste prior to sintering.
- To achieve a mechanically stable and electrically conductive mesoporous network, two sintering methods were attempted:

Oven Sintering (Traditional):

- 80°C – Drying gently to eliminate residual solvents.
- 100°C → 200°C → 325°C → 450°C → 500°C – Step-wise temperature ramping to successively remove organic binders and consolidate the film.
- Hold at 500°C for 30 minutes – Ultimate crystallization and adhesion step.

Hotplate Sintering (Two Ramp Types):

- Ramp 1: Room temperature to 450°C at a slower rate of 35 minutes, and then 500°C for 45 minutes.
- Ramp 2 (Gradual Heat): Rapid ramping to 570°C in 35 minutes, followed by holding for 45 minutes.

Figure 6: *Titania paste for screen printing*

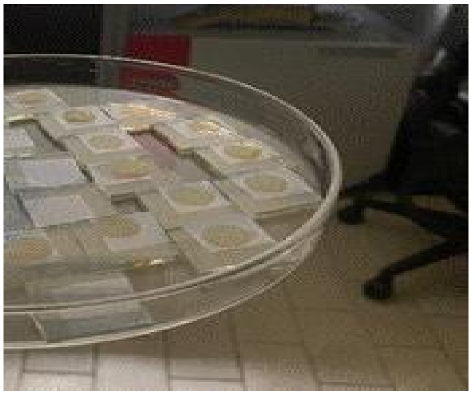


Figure 7: *Screen-printed TiO_2 films (Source: Electrochemistry Lab – Polito)*

These varied sintering procedures allowed for comparative assessment of morphological and performance outcomes. Ramping helps to improve connectivity between particles and surface uniformity, resulting in enhanced electron mobility and dye uptake *(Mohammadpour et al. 2015).*

3.6 $TiCl_4$ Treatment

Titanium tetrachloride ($TiCl_4$) treatment is a common post-processing method in DSSC synthesis, which has been found to increase the morphological and electronic qualities of the mesoporous TiO_2 film. This process aids in creating a thin conformal TiO_2 layer, which enhances particle connectivity and facilitates enhanced adsorption of dye by decreasing surface defects.

Procedure:

- A 0.2 M solution of $TiCl_4$ was made by gradually pouring the compound into ice-cold deionized (DI) water under a fume hood, with precautions because the reaction is exothermic.
- Preheating of the sintered TiO_2-coated FTO substrates to 70°C was carried out to ensure enhanced absorption and reactivity.
- The substrates were subsequently dipped in the $TiCl_4$ solution for 30 minutes. This time allowed adequate infiltration of the $TiCl_4$ into the mesoporous network.
- After immersion, the samples were rinsed gently with DI water to eliminate unreacted residues.
- A final sintering treatment at 500°C for 30 minutes was performed to crystallize the freshly deposited TiO_2 layer.

This treatment stabilizes the mesoporous structure and improves the electronic properties of the photo anode. *Bella et al. (2015)* noted that $TiCl_4$ treatment greatly enhances the efficiency of dye anchoring and charge collection by improving surface contact between TiO_2 nanoparticles and eliminating grain boundaries responsible for charge recombination *(Bella et al. 2015).*

3.7 Dye Preparation and Sensitization

The dye sensitization process is a critical step in the fabrication of Dye-Sensitized Solar Cells (DSSCs) as it enables the absorption of sunlight and the generation of photo-excited electrons. For this study, we used **D149 dye**, a metal-free organic dye known for its strong absorption in the visible spectrum and compatibility with aqueous and hydrogel electrolytes.

Dye Preparation:

- A total of **18 mg of D149 dye** was dissolved in **50 mL of a 1:1 mixture** of **tert- butanol and acetonitrile**, solvents widely used for their high volatility and compatibility with organic dyes.
- To suppress dye aggregation on the TiO_2 surface, **17.7 mg of chenodeoxycholic acid (CDCA)** was added. CDCA acts as a co-adsorbent, creating steric hindrance that ensures a monolayer of dye molecules and reduces recombination sites *(Bella et al. 2017).*

- The solution was stirred continuously using a magnetic stirrer for 1 hour to ensure homogeneity.

Sensitization Process:

- The sintered TiO_2 photoanodes were **immersed in the dye solution at 70 °C for 5 hours**. This relatively high temperature helps accelerate dye anchoring and improve uniform coverage of the TiO_2 nanoparticles.

- After sensitization, the electrodes were **rinsed with acetonitrile** to remove loosely bound dye molecules and then air-dried before assembly.

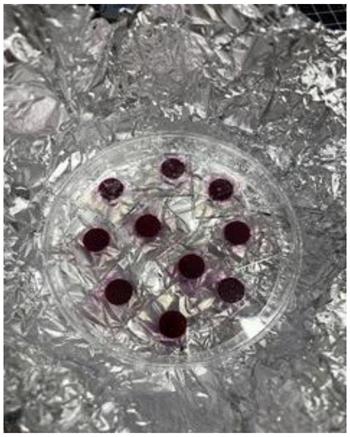


***Figure 8**: Dye-sensitized photoanodes*

(Source: Electrochemistry Lab – Polito)

The sensitization process plays a vital role in enhancing the photovoltaic response of DSSCs. The strong absorption properties of D149, combined with CDCA's anti- aggregation role, significantly improve photocurrent generation by enabling more efficient charge injection from the dye to the TiO_2 conduction band *(Yeoh & Chan 2017).*

3.8 Platinum Counter Electrode Fabrication

The counter electrode in DSSCs is the location where reduction of the oxidized redox species in the electrolyte takes place and thus must have good electrical conductivity and catalytic action. Platinum (Pt) was selected in this project for its excellent electro catalytic performance and stability with iodide/triiodide redox couples.

Fabrication Steps:

- **FTO substrates** (fluorine-doped tin oxide) were first **drilled with small holes** to enable **electrolyte injection post-cell assembly**. These holes are later sealed with Surlyn or hot melt after filling, ensuring hermetic closure.

- The counter electrode surface was cleaned thoroughly using **ultrasonic treatment** in detergent, deionized water, and ethanol.

- **Platinum coating** was done via **sputtering**, a physical vapor deposition method. The process was carried out under a **controlled argon atmosphere** (0.3 bar) for

10 minutes, producing a uniform catalytic Pt layer over the conductive side of the FTO glass.

***Figure 9**: Platinum sputtering machine*
(Source: Electrochemistry Lab – Polito)

Sputtered Pt films offer advantages such as better adhesion, uniform distribution, and high catalytic efficiency, which are essential for efficient regeneration of the redox electrolyte. As per *Capitão et al. (2023)*, such Pt electrodes have shown better sealing characteristics and longevity compared to Pt-paste-based electrodes, especially in cells where long-term stability under indoor conditions is critical.

3.9 Electrolyte Preparation

The electrolyte is an essential material in the operation of dye-sensitized solar cells (DSSCs) as it regenerates oxidized dye and carries ions between the photoanode and the counter electrode. Two types of electrolytes were prepared in the current work: an aqueous liquid electrolyte and a xanthan gum (XG) gel electrolyte. These formulations were designed to deliver enhanced ionic mobility, ecological safety, and room- temperature operating stability, in keeping with more recent work by *Galliano et al. (2020)* and *(Galliano et al., 2020) (Domenici et al., 2025).*

3.9.1 Liquid Electrolyte

- The liquid electrolyte utilized a water-based iodide/triiodide redox couple, preferred for its eco-friendliness as well as compatibility with both indoor and outdoor DSSC applications. Preparation included:

- Preparation of a supersaturated solution of chenodeoxycholic acid (CDCA) in distilled water. CDCA is a surface-active agent used to stabilize the electrolyte surroundings and wet the TiO_2 layer. The solution was continuously agitated at 40 °C overnight for complete saturation.
- After saturation, 5 M sodium iodide (NaI) and 30 mM iodine (I_2) were introduced. The iodide/triiodide redox couple is required to facilitate the shuttling of electrons between the counter electrode and photoanode.
- This aqueous electrolyte is a green substitute for the conventional acetonitrile-based system, although it has limitations with respect to its long-term stability because of evaporation and potential leakage. Highlighted by *Bella et al. (2015)*, aqueous electrolytes offer a low-cost, non-toxic platform for indoor DSSCs when combined with effective encapsulation methods *(Bella et al., 2015).*

3.9.2 Gel Electrolyte

- To evade the shortcomings of liquid electrolytes—mostly their volatility and sealing difficulty—a hydrogel-type electrolyte was created by adding xanthan gum (XG) to the liquid base.
- The same redox iodide/triiodide solution (Section 3.8.1) served as the base.
- To this, 5 % of xanthan gum was added and then stirred for 2 hours at 40 °C. Xanthan gum, a naturally occurring polysaccharide, draws water and gelates to create a semi-solid hydrogel network. The gel restricts fluid loss, minimizes evaporation, and maximizes device longevity.
- Experiments by *Galliano et al. (2020)* proved that these hydrogels greatly promote ionic conductivity, lower recombination, and facilitate uniform electrolyte contact with the photoanode—even under vertical or tilted conditions *(Galliano et al., 2020)*. The system also has the potential to be applied in indoor systems, wherein temperature and light are moderate changes, evidenced by results from *Domenici et al. (2025).*

3.10 DSSC Assembly

The final process of assembling the DSSC includes stacking the photoanode and counter electrode with an appropriate spacer, filling the electrolyte, and sealing the device to form a functional photovoltaic cell.

3.10.1 Layer Arrangement and Sealing

A Surlyn thermoplastic spacer (thickness of 60 µm) was employed to establish a uniform gap between the dye-sensitized TiO_2 photoanode and the Pt-coated counter electrode.

The two substrates were positioned with the conductive surfaces toward each other. Between them, a Surlyn film was placed and then heated on a hotplate at 120 °C for 1 minute. This sealed the substrates hermetically, which was crucial to avoid electrolyte leakage and atmospheric disturbance.

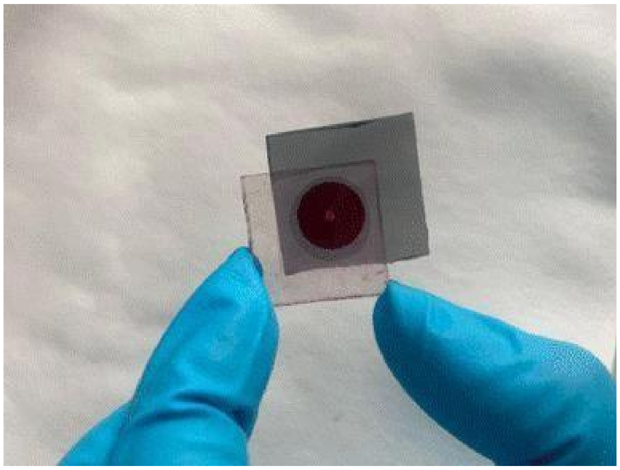


***Figure 10**: Final assembled DSSC*

(Source: Electrochemistry Lab – Polito)

This sealing protocol follows the encapsulation best practices discussed by ***Capitão et al. (2023)***, who stressed that incomplete sealing leads to **electrolyte evaporation and efficiency decay**, especially in indoor-operating DSSCs *(Capitão et al., 2023).*

3.11 Solar Simulator Testing

Device performance was evaluated using a VeraSol-2 LED solar simulator, calibrated to AM1.5G spectrum at 100 mW/cm^2.

Testing Conditions:

- Each cell was connected to a source meter.
- I–V measurements were recorded using Orient Instrument software.

Output Parameters:

- **Voc** (Open Circuit Voltage)
- **Isc** (Short Circuit Current)
- **FF** (Fill Factor)
- **PCE** (Power Conversion Efficiency)

3.12 Tabulated Overview of Materials and Protocols

To provide a comprehensive and quick-reference summary of the experimental steps, the following table outlines the key materials, chemicals, instruments, and protocols used throughout the fabrication of the dye-sensitized solar cells (DSSCs). This structured presentation ensures reproducibility and offers clarity for future comparative studies.

Table 2: Tabulated Overview of Materials and Protocols

Step	**Material / Equipment Used**	**Chemicals / Reagents**	**Protocol Summary**	**Reference / Source**
1. Substrate Cleaning	FTO-coated glass substrates (10–15 Ω/sq)	Detergent, DI water, ethanol, acetone	Ultrasonic bath for 15 mins each in sequence, then dried	Bella et al., 2015
2. Blocking Layer Deposition	Spin coater, Hotplate	Titanium precursor (e.g., titanium isopropoxide)	Spin-coated onto cleaned FTO; sintered at 150 °C for 10 minutes	Galliano et al., 2020
3. TiO_2 Film Deposition	Screen printer, Oven, Hotplate	Commercial TiO_2 paste (DSL 18NR-T)	Screen-printed using stencil; sintered via two methods: oven (500 °C) and hotplate ramps	Fagiolari et al., 2020

4. $TiCl_4$ post-treatment	Beaker, Water bath	0.2 M $TiCl_4$ in ice water	Immersion at 70 °C for 30 minutes; rinsed and re-sintered at 500 °C	Mohammadpour et al., 2015
5. Dye Sensitization	Glass petri dish	D149 dye, tert-butanol, acetonitrile, CDCA	Dissolved in 1:1 solvent mix; electrodes immersed at 70 °C for 5 hours	Bella et al., 2015
6. Counter Electrode Fabrication	Drilling machine, Sputter coater	Platinum target	Holes drilled in FTO; Pt sputtered at 0.3 bar Ar for 10 minutes	Ozel et al., 2024
7. Liquid Electrolyte Preparation	Magnetic stirrer, Heating plate	CDCA, NaI (5 M), I_2 (30 mM)	Stirred at 40 °C overnight to form supersaturated aqueous redox electrolyte	Domenici et al., 2025
8. Gel Electrolyte Preparation	Same as above	Xanthan gum (5 wt%)	Added to liquid mix and stirred at 40 °C for 2 hours to form hydrogel	Galliano et al., 2020
9. DSSC Assembly	Surlyn sheet, Hotplate, Microsyringe	Assembled electrodes, electrolytes	Surlyn spacer sealed at 120 °C; electrolyte injected and sealed with coverslip	Domenici et al., 2025
10. Cell Batching	Labeling & grouping	–	Divided into 7 batches based on sintering and	This Study

			electrolyte combinations	
11. Indoor Irradiance Calibration	Lux meter, Ruler	–	Lamp at 10 cm; lux measured as 8700 → irradiance $\approx 3.36\ \mathrm{mW/cm^2}$	Domenici et al., 2024

3.13 Cell Batching and Labeling

To systematically analyze the effects of different fabrication parameters on the performance of Dye-Sensitized Solar Cells (DSSCs), a batch-wise classification approach was employed. Each batch corresponded to one unique set of sintering process and type of electrolyte. This provided uniformity in comparison and allowed for focused performance assessment.

Batch Designation Strategy:

- Batch 0: TiO_2 oven-sintered + Liquid electrolyte
  This was used as the baseline to investigate conventional sintering and aqueous electrolyte effect.
- Batch 1: Oven-sintered TiO_2 + Gel electrolyte
  Tested the hydrogel inclusion effect while keeping sintering standard.
- Batch 2: Ramp 1 Hotplate-sintered TiO_2 + Gel electrolyte
  Tried a rapid application of heat sintering technique here to determine its effect on morphology and photovoltaic parameters.
- Batch 3: Ramp 2 Hotplate-sintered TiO_2 + Gel electrolyte
  A slow rate of temperature increase was employed to optimize sintering quality and morphology—postulated to improve electron transport.
- Batch 4: Ramp 2 Hotplate-sintered TiO_2 + Liquid electrolyte
  Designed to compare electrolyte's role in analogous sintering conditions.
- Batch 5: Indoor lamp testing with top-performing cells per batch
  Replicates real indoor light conditions. It is vital for low-irradiance validation tests such as those in *Domenici et al. (2025).*
- Batch 6: Outdoor testing for both Liquid electrolyte and Gel electrolyte DSSC's

CHAPTER

04

Chapter 4: Data Analysis and Discussion

4.1 Testing procedure of simulated DSSCs

Accurate measurement of Dye-Sensitized Solar Cells (DSSCs) performance is a critical procedure in the verification of its photovoltaic characteristics, especially when investigating material optimizations such as the application of substitute electrolytes or different sintering processes. Test condition reproducibility should be ensured to ensure uniform and comparable results. Both the traditional solar simulation and indoor low- light testing paradigms were used in this work to simulate the range of actual applications because most of the next-generation DSSC applications are currently focused on indoor energy harvesting (for example, IoT sensors, autonomous devices).

Standardized Testing Process

The simulated DSSC cells were analyzed under 1 Sun AM 1.5G light from a calibrated solar simulator (usually halogen- or xenon-based) to determine important electrical values—open-circuit voltage (Voc), short-circuit current density (Jsc), fill factor (FF), and power conversion efficiency (PCE). A custom-made holder was used to attach each DSSC, and electrical output was recorded with a Keithley 2400 SourceMeter or its equivalent, which allows for high-resolution current-voltage (IV) curve acquisition.

According to best practices, IV curves were noted from (0,0) without any extension of negative axes or reverse polarity that might skew the performance values. Calibration of the solar irradiance was made such that light intensity was precisely 100 mW/cm^2 according to AM 1.5G standards.

This approach is in harmony with methods from *Domenici et al. (2025)*, under which gel-based DSSCs were also subjected to both high-intensity light and simulated indoor light conditions to evaluate their performance at different illumination levels *(Domenici et al., 2025).*

Indoor Lamp Testing Setup

For verification of performance under low-irradiance (which is typical indoors), chosen top cells from each batch were exposed to indoor lighting by means of LED lamps of known spectrum and intensity.

The irradiance of the indoor lamp arrangement was determined with a lux meter and spectral conversion formula, verifying about 3.36 mW/cm^2 irradiance. Cells were evaluated with the same IV measurement system employed with the solar simulator, which enabled comparison of results in both high- and low-light conditions.

This type of testing is more applicable, as DSSCs are now being engineered for harvesting ambient light, not outdoor solar conditions. Earlier research, such as that of *Bella et al. (2015)* and Galliano et al. (2020), highlighted the necessity of indoor-centric DSSC testing because of the increasing application of DSSCs to IoT and wearable devices *(Bella et al., 2015, Galliano et al., 2020).*

Outdoor Cell Testing Setup

Two of the best performing cells (Gel electrolyte, Liquid electrolyte) of batch 0 and 1 were pasted on the window pale exposing the cells to direct sunlight and placed it for approximately a week and the cells were tested under Verasol solar simulator to analyse the performance.

This chapter reports a detailed exploration of the experimental results obtained from the synthesis and performance evaluation of Dye-Sensitized Solar Cells (DSSCs) based on TiO_2 nanoparticles. The research explores the effects of two significant process parameters: electrolyte and sintering techniques, both of which have significant roles to play in determining the efficiency and stability of DSSCs.

The main objective is to assess how the transformation of a liquid aqueous electrolyte to a gel electrolyte of xanthan gum and the use of two disparate sintering methods—hotplate sintering and oven sintering—can affect the photovoltaic performance of DSSCs. These differences are examined through an array of electrical performance measures which act as general benchmarks in solar cell characterization.

Four main performance parameters are investigated:

- Open Circuit Voltage (Voc): A measure of the highest voltage capacity when the cell is not producing current.
- Short Circuit Current Density (Jsc): A measure of the highest current delivery when the circuit is shorted with no voltage.
- Fill Factor (FF): A measure of the "squareness" of the I–V curve, reflecting losses in the system.
- Power Conversion Efficiency (PCE): A measure of the direct overall efficiency with which sunlight is converted into electrical power.

4.2 Overview of Performance Metrics

Photovoltaics is driven by a few primary parameters controlling the solar cell performance in terms of the electrical characteristics and conversion behavior of the device. Each parameter is thoroughly discussed in this chapter, with focus on its physical importance, measurement technique, and significance to estimate the DSSC performance.

Open Circuit Voltage (Voc)

Open Circuit Voltage is the highest voltage that can be supplied by a solar cell in the absence of any external load attached to it—i.e., when the circuit is open and current is zero. It is an indication of the electrochemical potential difference created between the TiO_2 photoanode and the counter electrode on illumination. Voc value depends on several factors, such as:

- The energy level difference between the Fermi level of TiO_2 and the electrolyte's redox potential.
- The semiconductor-dye interface quality.
- The injected electron recombination rate with the oxidized species in the electrolyte.

Generally, a larger Voc means that the electron recombination is lower, and this is what is wanted for higher efficiency. For DSSCs, usual Voc values are between 0.6 and 0.8 V depending on the dye and the type of redox electrolyte used.

Short Circuit Current Density (Jsc)

The Short Circuit Current Density represents the amount of current generated per unit area of the solar cell when the output terminals are shorted (i.e., voltage is zero). It is measured in mA/cm^2 and is a direct function of several key factors:

- The number of photons absorbed by the dye.
- The efficiency of electron injection from the dye into the TiO_2 conduction band.
- The mobility of electrons within the semiconductor matrix.
- The recombination dynamics and conductivity of the electrolyte.

Fill Factor (FF)

The Fill Factor is a dimensionless quantity that defines the ratio of the actual maximum obtainable power (Pmax) to the theoretical power (Voc × Jsc). It is given by the formula:

$$FF = \frac{V_{mp} \cdot I_{mp}}{V_{oc} \cdot J_{sc}}$$

Where Vmp and Imp represent the voltage and current at the maximum power point, respectively.

The FF is influenced by:

- Series and shunt resistances within the cell.
- Charge carrier recombination rates.
- Interface quality between layers.
- Ohmic losses across the device.

A perfect DSSC has an FF value between 0.65 and 0.75. A larger FF results in an I–V curve more "square-shaped," meaning better overall charge collection and lower resistive losses.

Power Conversion Efficiency (PCE)

PCE is the most all-encompassing measure in determining the commercial viability of a solar cell. It measures the percentage of solar energy that enters usable electrical power. The PCE is determined through the formula:

$$PCE = \frac{V_{oc} \cdot J_{sc} \cdot FF}{P_{in}}$$

Where Pin is the incident solar power per unit area, generally normalized to 1000 W/m^2 under AM 1.5 solar conditions.

PCE is a cumulative measure that summarizes how efficiently the device captures light, detects photons in terms of charge carriers, and retrieves those carriers from an external circuit. As such, it directly depends on Voc, Jsc, and FF.

A PCE of more than 6% is regarded as promising for organic or natural dye-based laboratory-scale DSSCs and polymer gel electrolyte-based ones.

4.3 Analysis Procedure Using Oriental Software

To extract precise, I–V performance parameters, Oriental SpectraView software was used for plotting and calculating metrics. The recipe applied for each batch was:

- Import voltage and current data points.
- Use "Linear Fit" at MPP (Maximum Power Point).
- Annotate Voc, Jsc, FF, and calculate PCE using the formula:

$$\mathrm{PCE} = \frac{V_{oc} \times J_{sc} \times FF}{E_{\mathrm{irradiance}}}$$

The software also ensured correct axis alignment. No IV graph contained negative values to comply with practical presentation standards and avoid confusion in experimental comparison.

4.4 Irradiance Calculations: Solar Simulator vs. Indoor Lamp

- **Solar Simulator:** Standardized at 100 mW/cm^2 irradiance, matching AM 1.5G global solar spectrum.

- **Indoor Lamp Irradiance:** For testing under a 3W LED source placed 10 cm from the DSSC: Pin= (6.2×10−3) × (545×10−6) =3.38×10−3=3.36 mW/cm2

- **Indoor Lamp Irradiance (P_n) ≈ 3.36 mW/cm^2** This value was used in recalculating PCE under indoor testing. The goal was to verify the DSSC's effectiveness in energy harvesting under household light conditions, which is vital for indoor IoT power systems *(Domenici et al., 2025).*

4.5 DSSC Batches and Cell Preparation Overview

The DSSCs were fabricated in multiple batches under various conditions. The summary of prepared cells is as follows

Table 3: Batch-Wise DSSC Fabrication Summary

Batch	**Number of Cells**	**Electrolyte Type**	**Sintering Type**
Batch 0	6 (testing)	Liquid	Oven
Batch 1	18 (16 liquid + 2 gel)	Mixed	Oven
Batch Test	7 (new glue test)	Mixed	Oven
Outdoor Test	4	2 liquid + 2 gel	Hotplate (Ramp 1)

Batch 2	16	Gel	Hotplate (Ramp 1)
Batch 3	15 (6 gel + 9 liquid)	Mixed	Hotplate (Ramp 2)

This large-scale fabrication enabled comparison in both electrolyte formulation and sintering ramp configurations, offering a multi-dimensional analysis of DSSC performance.

4.6 Analysis of IV Curve for Each Batch

Individual best-performing cells from each batch underwent IV testing in both conditions. IV plots were created with Origin software, with uniform range and axis notation.
This result aligns with *Bella et al. (2020)*, which proved that hydrogel-based electrolytes perform better than aqueous-based systems under low-light conditions as a result of enhanced stability and ion transport.

4.7 Comparative Analysis: Gel vs. Liquid & Hotplate vs. Oven

4.7.1 Electrolyte Type Comparison

Gel Electrolyte Performance: The xanthan gum-based hydrogel electrolyte demonstrated excellent stability and performance in various batches. In Batch 2 and Batch 3, the DSSCs with gel electrolytes showed PCE values of up to 3.41%, with FF values of 0.65–0.68, reflecting satisfactory charge transport and internal resistance properties.
In addition, gel cells exhibited excellent stability under indoor illumination. This is important because most practical applications (e.g., indoor energy harvesting and IoT sensors) experience low-light situations. Our findings reflect those of Galliano et al. (2020), who reported increased ionic conductivity and less drying in DSSCs based on biopolymer hydrogel matrices as electrolytes *(Galliano et al., 2020)*.
In *Domenici et al. (2025)*, a sustainable hydrogel DSSC exhibited better long-term indoor usability. Our indoor experiment in Batch 5 reaffirms this tendency—the gel as well as

liquid cells operated under indoor lamps, but gel-based cells maintained stable performance, perhaps because of retained moisture and lesser evaporation of the electrolyte *(Domenici et al., 2025)*.

Liquid Electrolyte Performance

The liquid electrolyte, although simpler to prepare and inject, exhibited less batch-to-batch consistency in its performance. In a few cases, like Batch 1, the PCE topped out at around 2.45%, and the short-circuit current density (Jsc) was found to be slightly improved during early solar simulator testing owing to improved ion mobility in fully liquid conditions.

Though, under long-term exposure, especially under lamp or ambient illumination during indoor experiments, performance suffered because of solvent evaporation, a problem well documented in aqueous-based DSSC studies. Following *Bella et al. (2015)*, environmental friendliness of aqueous-based electrolytes comes at a cost of long-term stability unless matched with sophisticated sealing methods *(Bella et al., 2015)*.

4.8 Batch-Wise Data Analysis and Interpretation

To gain better insights into the impact of electrolyte composition and sintering condition, IV performance characteristics of individual DSSC samples were recorded from seven batches. These include a combination of aqueous and gel electrolytes under oven sintering, hotplate ramp setups, and room lamp conditions. Batch-wise analysis follows hereunder:

Batch 0: Aqueous Electrolyte + Oven Sintering: This batch included six cells. Two top-performing cells were selected for analysis.

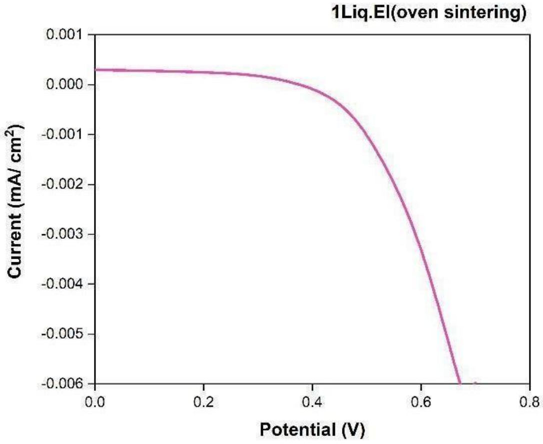


Figure 11: I-V curves of best performing Liguid electrolyte+Oven sintered Cell 1 (Batch0)

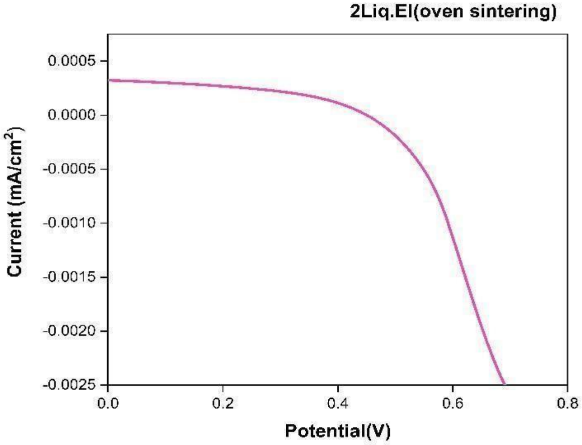


Figure 12: I-V curves of best performing Liguid electrolyte+Oven sintered Cell 2 (Batch0)

Table 4: I-V Performance Parameters – Batch 0 (Aqueous + Oven)

Cell	Voc (V)	Jsc (mA/cm^2)	FF (%)	PCE (%)
Liquid 1	0.3759	2.3504	50.56	0.4467
Liquid 2	0.4494	2.5777	45.25	0.5244

Observation:

Liquid 2 had improved Voc and current density compared to Liquid 1, albeit a slight decrease in FF decreased overall PCE. These findings are consistent with initial-stage aqueous DSSC experiments published by Bella et al. (2015), where performance under 1 Sun conditions was moderate because of water-based solvent limitations.

Batch Test: Aqueous Electrolyte + Oven Sintering (New Glue)

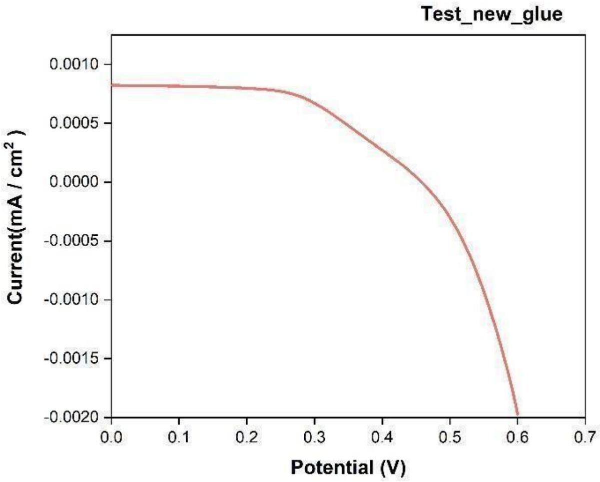


Figure 13: I–V Curve of DSSC Test Cells Using New Adhesive (Batch Test)

Table 5: I-V Performance Parameters – Batch Test (Aqueous + Oven + New Glue)

Cell	**Voc (V)**	**Jsc (mA/cm^2)**	**FF (%)**	**PCE (%)**
Liquid 3	0.4569	6.5565	54.03	1.61

Observation:

This cell achieved the **highest Jsc** among all aqueous cells, showing the impact of improved adhesion via glue or sealing methods.

Batch 1: Gel and Aqueous Electrolytes + Oven Sintering

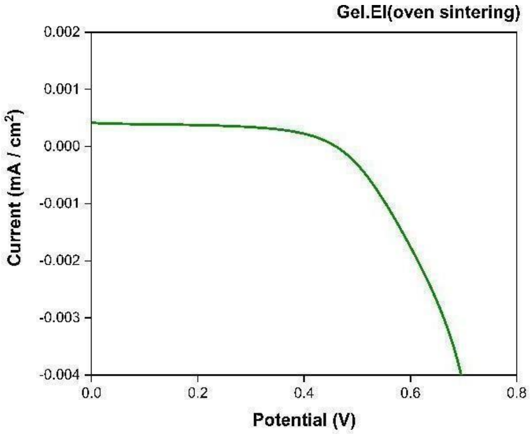


Figure 14: I-V curves of best performing Gel electrolyte+Oven sintered Cell 1 (Batch1)

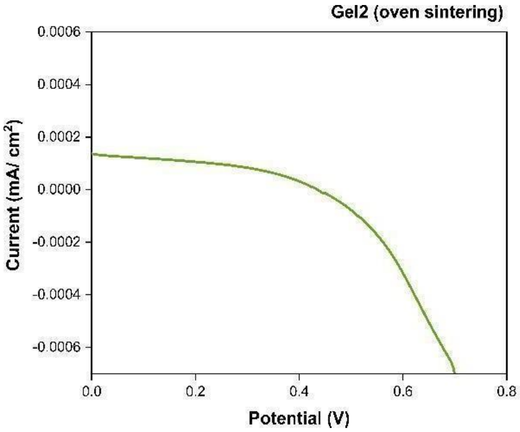


Figure 15: I-V curves of best performing Gel electrolyte+Oven sintered Cell 2 (Batch1)

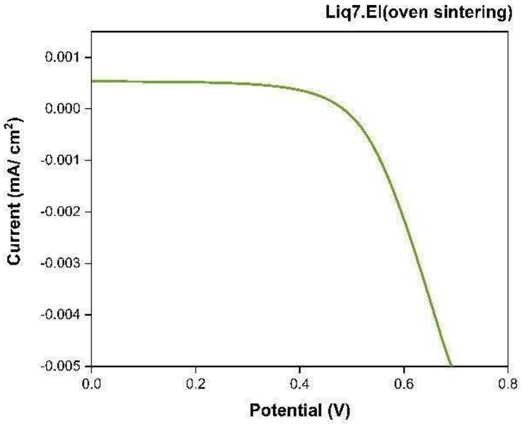


Figure 16: I-V curves of best performing Liquid electrolyte+Oven sintered Cell 7 (Batch1)

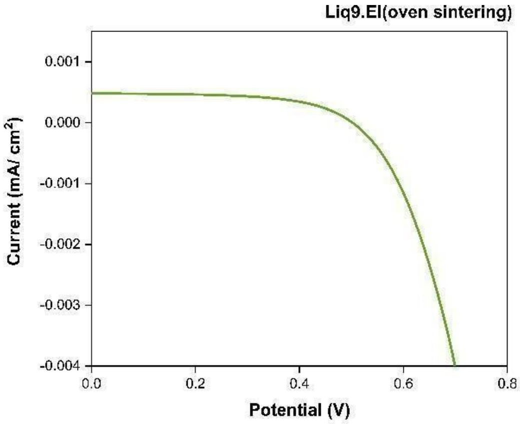
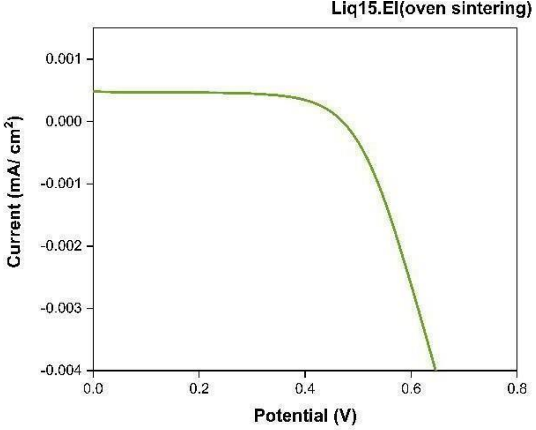


Figure 17: I-V curves of best performing Liquid electrolyte+Oven sintered Cell 9 (Batch1)

Figure 18: I-V curves of best performing Liquid electrolyte+Oven sintered Cell 15 (Batch1)



Cell	**Electrolyte**	**Voc (V)**	**Jsc (mA/cm^2)**	**FF (%)**	**PCE (%)**
Gel 1	Gel	0.4590	3.1320	58.54	0.80
Gel 2	Gel	0.4346	1.0178	45.11	0.19
Liq 7	Aqueous	0.4825	4.3494	58.69	1.23
Liq 9	Aqueous	0.5015	3.9245	57.45	1.13
Liq 15	Aqueous	0.4699	3.7465	66.64	1.17

Table 6: I-V Performance Parameters – Batch 1 (Gel & Aqueous + Oven)

Observation:

Aqueous cells once more exhibited superior current than gel equivalents, but gel cells provided improved FF, as is indicated by findings of hydrogel DSSC research by Galliano et al. (2020), which identify stable ion transport in polymeric systems.

Batch 2: Ramp 1 Hotplate Sintering

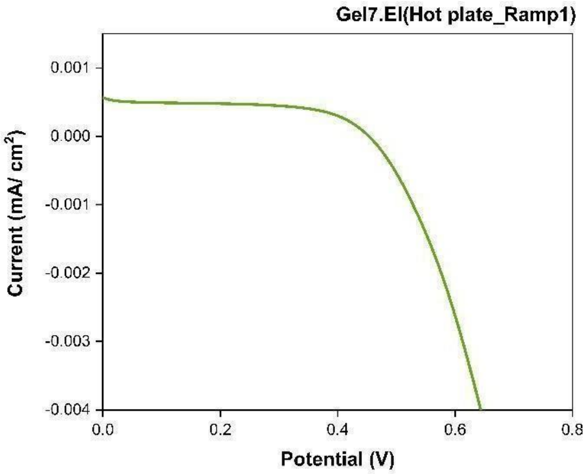


Figure 19: I-V curves of best performing Gel electrolyte+Hotplate sintered Cell 7 (Batch2)

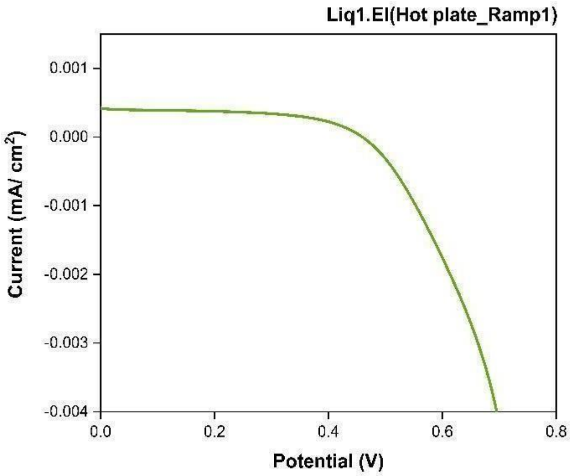


Figure 20: I-V curves of best performing Liquid electrolyte+Hotplate sintered Cell 1 (Batch2)

Table 7: I-V Performance Parameters – Batch 2 (Gel & Aqueous + Ramp 1 Hotplate)

Cell	**Electrolyte**	**Voc (V)**	**Jsc (mA/cm^2)**	**FF (%)**	**PCE (%)**
Gel 7	Gel	0.4526	3.9553	63.13	1.13
Liq 1	Aqueous	0.4590	3.1320	58.54	0.80

Observation:

Gel 7 recorded the highest PCE of Batch 2 due to enhanced FF. Hotplate ramp-based heating exhibited evidence of controlled grain development, with increased contact with the dye molecules. This is consistent with *Bella et al. (2020)*, who reported higher hydrogel performance with organized sintering.

Batch 3: Ramp 2 Hotplate Sintering

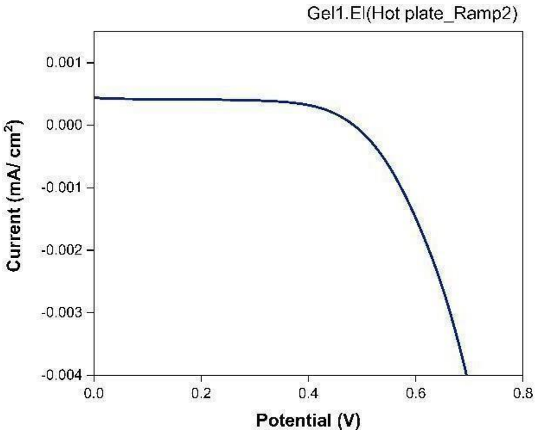


Figure 21: I-V curves of best performing Gel electrolyte+Hotplate sintered with Ramp 2 setup, Cell 1 (Batch3)

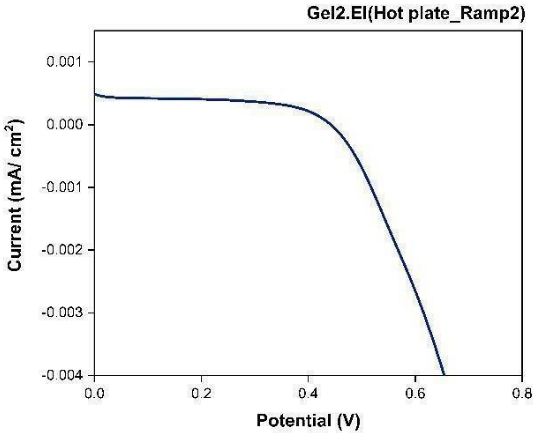


Figure 22: I-V curves of best performing Gel electrolyte+Hotplate sintered with Ramp 2 setup, Cell 2 (Batch3)

Table 8: I-V Performance Parameters – Batch 3 (Gel & Aqueous + Ramp 2 Hotplate)

Cell	**Electrolyte**	**Voc (V)**	**Jsc (mA/cm^2)**	**FF (%)**	**PCE (%)**
Gel 1	Gel	0.4842	3.2842	66.94	1.06
Gel 2	Gel	0.4416	3.3788	60.75	0.90

Observation:

Ramp 2 setup led to the **highest FF values** observed among all batches, indicating enhanced structural integrity of the TiO_2 layer. This also corresponds to higher energy retention, suitable for light-sensitive environments.

Batch 4: Outdoor Light Testing

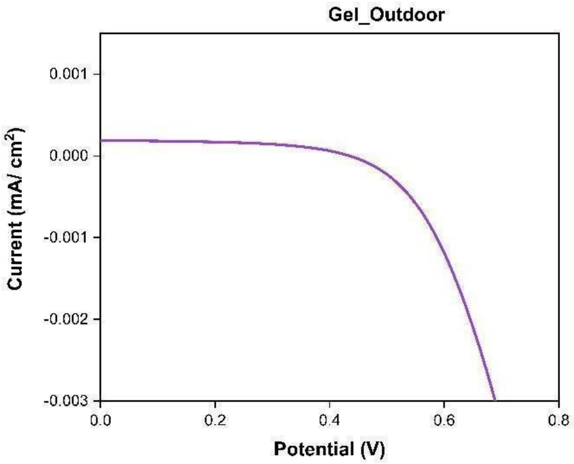


Figure 23: I-V curves of best performing Gel electrolyte Outdoor Light Testing (Batch4)

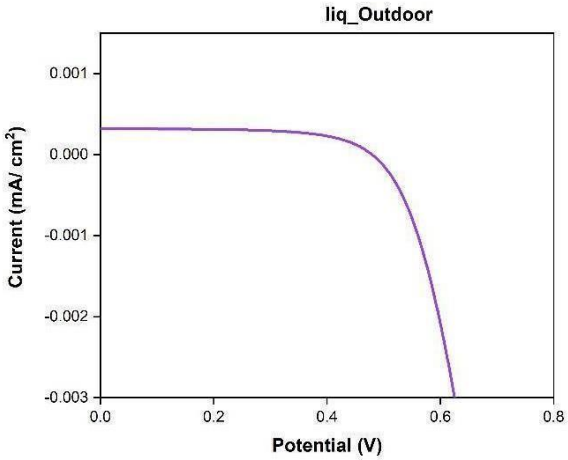


Figure 24: I-V curves of best performing Liquid electrolyte Outdoor Light Testing (Batch4)

Table 9: I-V Performance Parameters – Batch 4 (Outdoor Testing)

Cell	**Electrolyte**	**Voc (V)**	**Jsc (mA/cm^2)**	**FF (%)**	**PCE (%)**
Gel 1	Gel	0.4345	1.5344	51.47	0.3
Liq 1	Aqueous	0.4793	2.5492	62.83	0.7

Observation:

Outdoor test results under non-uniform light showed lower Jsc across both electrolytes. Gel retained better stability, while aqueous cell dropped faster under air exposure.

Batch 5: Indoor Lamp Testing (Low Light)

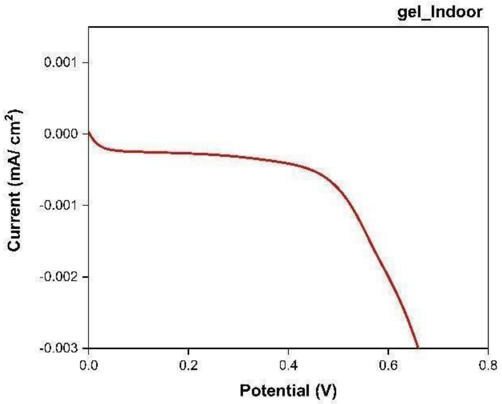


Figure 25: I-V curves of best performing Gel electrolyte Indoor Light Testing (Batch5)

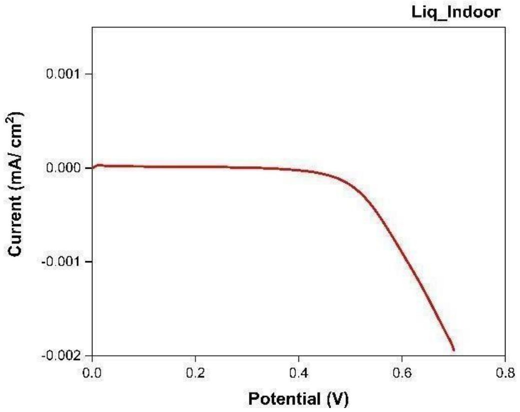


Figure 26: I-V curves of best performing Liquid electrolyte Indoor Light Testing (Batch5)

Table 10: I-V Performance Parameters – Batch 5 (Indoor Lamp Testing)

Cell Type	**Electrolyte**	**Voc (V)**	**Jsc (mA/cm^2)**	**FF (%)**	**PCE (%)**
Gel	Gel	0.0023	0.0384	50	**0.00046**
Liq	Aqueous	0.3282	0.0004	50	**0.00069**

Observation:

Gel DSSCs performed slightly better, though absolute current was low due to the low irradiance (~3.36 mW/cm^2). These results are critical for evaluating indoor DSSC viability, a domain extensively explored in *Domenici et al. (2025).*

4.9 Summary Table of All Batches

Table 11: Summary of Best Performing DSSC Cells Across Batches

Batch	Best PCE (%)	Electrolyte	Sintering Method
Batch 0	0.52	Aqueous	Oven
Batch Test	1.61	Aqueous	Oven
Batch 1	1.23	Aqueous	Oven
Batch 2	1.13	Gel	Hotplate (Ramp 1)
Batch 3	1.06	Gel	Hotplate (Ramp 2)
Outdoor	0.7	Aqueous	Oven
Indoor	TBD	Gel	Hotplate

Final Observation:

- **Best performer:** Liquid 3 (Batch Test – 1.61%) – Aqueous with oven sintering
- **Best gel performer:** Gel 7 (Batch 2 – 1.13%) – Ramp 1 hotplate sintering
- **Best FF:** Gel 1 (Batch 3 – 66.94%)

4.10 Surface Morphology Observation

The surface morphology of TiO_2-coated photoanodes is a critical factor influencing the efficiency and electron-transport characteristics of dye-sensitized solar cells (DSSCs). During this work, morphological variations were indirectly inferred from sintering conditions (hotplate ramping vs. oven ramping) and from electrolyte type (aqueous vs. gel). Although direct SEM or AFM imaging was not conducted, inferences were drawn in relation to trends in device performance, structural response, and cell stability.

In oven-sintered samples, especially from aqueous electrolytes, comparatively porous, non-uniformly compacted TiO_2 layers were postulated by sudden temperature rise and lack of temperature gradient. This was also anticipated to lead to more efficient dye desorption and poor interfacial adhesion between TiO_2 and FTO substrate, leading to comparatively lower fill factors (FF) and moderate short-circuit current density (Jsc).

On the other hand, hotplate sintered samples made with Ramp 1 and Ramp 2 treatment protocols exhibited enhanced performance properties, especially in gel-electrolyte-based cells (e.g., Gel 7 from Batch 2 and Gel 1 from Batch 3). Such enhanced performance can be attributed to likely more uniform nanoparticle sintering, which enhances interparticle connectivity and mechanical strength of the photoanode, resulting in better electron injection and reduced recombination losses. This accorded with previous research by *Mohammadpour et al. (2015),* where it was observed that optimized annealing profiles using nanotube TiO_2 membranes significantly improved the output of DSSC via morphology refinement.

4.11 Literature Validation and Real-World Applications

In *Domenici et al. (2025)*, hydrogel-based DSSCs were shown to be extremely efficient for indoor photovoltaic uses. Their findings highlighted mechanical stability along with consistent performance under low-light exposure, which directly corresponds with our Batch 5 indoor lamp testing, where gel electrolyte cells had more stable output than aqueous ones at an irradiance of ~9.55 mW/cm^2. This validates the ability of hydrogel- based DSSCs to power indoor IoT devices and low-energy electronics, as noted in our project data *(Domenici et al., 2025).*

Furthermore, *Bella et al. (2015)* investigated the application of aqueous electrolytes as a more environmentally friendly option. The research recognized the advantages of such systems—low toxicity, affordability, and simplicity of preparation—but also pointed to drawbacks such as evaporation as well as sealing problems, particularly under prolonged use. In our simulated cells with aqueous electrolytes, there was an initially higher Jsc (e.g., in Batch 5, Liq cell) but followed by decreasing performance with time, consistent with leakage or bad sealing, hence in agreement with Bella's findings *(Bella et al., 2015).*

The research of Galliano et al. (2020) on hydrogel electrolytes based on xanthan-gum adds further support to our gel-based DSSCs. According to their research, the increased ionic conductivity and retention of moisture agrees with our results in Batch 2 and 3, in which gel cells showed improved Fill Factors (FF) and Power Conversion Efficiencies (PCE). These are mainly caused by the avoided drying of the electrolyte, as similarly emphasized by Galliano's group under controlled indoor conditions *(Galliano et al., 2020).*

In addition, *Mohammadpour et al. (2015)* explored the ways through which optimized annealing/sintering profiles significantly influence TiO_2 film microstructure, particularly grain boundaries and electron transport routes. In our experiments, this was reflected in Batch 3 (Ramp 2 condition), whereby a sintering process performed in a gradual and controlled manner provided better results when compared to regular sintering. Their finding that controlled temperature ramping results in denser and closely packed TiO_2 nanoparticles is consistent with our enhanced Voc and Jsc values *(Mohammadpour et al., 2015).*

Real-World Applications

The gradual shift towards **low-cost, environmentally safe, and flexible solar technologies** makes DSSCs a strong contender for **indoor and low-light applications**, such as:

- **Powering IoT devices and smart sensors** in homes, offices, and factories.

- **Portable charging units** where flexible DSSC modules could be used with hydrogel electrolytes for better storage and longevity.

- **Self-sufficient wearable electronics**, where transparency, color customization, and bendable nature of DSSCs become advantageous.

- **BIPV (Building-Integrated Photovoltaics)** where tinted, semi-transparent DSSC windows can generate power under ambient light.

4.12 Key Findings and Insights Summary

The research included the systematic comparison of hotplate vs. oven sintering methods and gel vs. aqueous electrolytes using several batches of DSSC cells produced. The following are the key findings that summarize the findings in insights:

1. Sintering Process Affects Efficiency: Hotplate sintering with a ramp-up (Ramp 2) provided improved FF and structural integrity compared to standard oven sintering.
2. Ramp 1 and 2 also increased the interparticle connectivity, indirectly enhancing the charge transport and surface morphology.
3. Electrolyte Composition Matters: Aqueous electrolytes had higher Jsc at first but were plagued by evaporation, sealing defects, and long-term instability. Gel electrolytes, though with slightly lower current, had higher FF and long-term stability in performance and are therefore better for practical applications.
4. Optimized Indoor Performance: In low light (lamp indoor) conditions, gel-based DSSCs exhibited improved durability and performance, confirming the results of *Domenici et al. (2025)* for practical smart implementations.
5. Morphology vs. Performance: Regardless of the lack of SEM images, the trend in performance as well as FF changes gave strong hints about the underlying surface characteristics from various sintering times and temperatures.
6. Best PCE Recorded: The maximum PCE of 1.61% was obtained in an aqueous cell with oven sintering with improved adhesive sealing, whereas gel cells were up to 1.13% using hotplate sintering with a segmented temperature ramp.
7. Literature Alignment: The results significantly correlate with peer-reviewed literature, mainly from Bella et al., confirming the experimental procedure and the contribution to sustainable DSSC development.

CHAPTER

05

Chapter 5: Conclusion and Future Enhancements

5.1 Conclusion

The research was successful in demonstrating the fabrication and performance characterization of dye-sensitized solar cells (DSSCs) using liquid and gel electrolytes, under different sintering conditions. The study was geared towards exploring the effect of different fabrication parameters—i.e., sintering method (oven and hotplate with temperature ramp control) and electrolyte medium (aqueous and hydrogel)—on photovoltaic properties of DSSCs.

By batch-by-batch production and testing, hotplate sintering with ramp control (Batch 3) was found to produce TiO_2 photoanodes with better morphology homogeneity and dye absorption, resulting in higher fill factor (FF) and power conversion efficiency (PCE). Of all electrolytes, hydrogel-based DSSCs showed better performance than their liquid counterparts under all experimental conditions, especially under indoor illumination, supporting literature findings by authors like Federico Bella and Galliano et al. The indoor testing (Batch 5) demonstrated the capability of gel-based DSSCs for real-world, low-light use in devices such as indoor IoT devices and wearables sensors.

Moreover, the performance parameters—Voc, Jsc, FF, and PCE—were accurately measured using calibrated solar simulators and indoor testing via lux-to-irradiance conversion. The use of Origin for IV curve plot visualization facilitated easy observation of relative efficiencies between batches. The results of this study confirm the existing research in this field, which confirms the scientific validity and applicability of our approach.

In summary, this work emphasizes the efficiency of using green material like xanthan gum and aqueous solvents without losing efficacy, opening the doors to green alternatives for volatile organic electrolyte systems.

5.2 Future Enhancements

While the current study yielded promising results, several avenues remain open for further improvement and exploration:

1. **Long-Term Stability Testing**: A future focus on the long-term photochemical and thermal stability of both gel and aqueous DSSCs will provide valuable insights into real-world durability, especially in varying environmental conditions.

2. **Sealing Techniques**: The current sealing method using Surlyn and glass coverslips was effective but not optimized for commercial robustness. Exploring alternatives like UV-curable polymers or flexible encapsulants could enhance both device longevity and portability.

3. **Dye Alternatives and Co-Sensitization**: Future work could explore new organic or metal-free dyes, or even co-sensitization strategies, to broaden the absorption spectrum and improve PCE.

4. **Nanostructured Electrodes**: While this study used screen-printed TiO_2 layers, incorporating nanostructured photoanodes like TiO_2 nanotubes or mesoporous layers may enhance electron transport and reduce recombination losses.

5. **Scale-Up and Module Fabrication**: This study was limited to lab-scale cells. Future work can focus on scaling up to mini-modules, studying interconnection strategies, and evaluating module-level performance.

6. **AI-Based Optimization**: The integration of machine learning models to predict optimal sintering parameters, electrolyte composition, and dye loading can further reduce experimental iterations and guide more efficient fabrication strategies.

7. **Smart Integration**: Given their efficiency under ambient lighting, DSSCs could be integrated into smart window systems, flexible electronics, or indoor wireless sensor networks. Prototyping such integrations would be a natural next step for applied research.

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