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# Experimental analysis of the waste hydrothermal treatment process

**English Summary of** 

Ph.D. thesis of

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## 1 General Introduction

The first chapter of this doctoral thesis lays the foundation for understanding the complex challenges of modern waste management in a global context. In recent decades, the world has witnessed an unprecedented rise in the production and consumption of plastic materials, fiber-reinforced composites, and electronic-based products. This surge has been driven by industrial growth, expanding populations, urbanization, and the increasing deployment of renewable energy infrastructure. While these materials have facilitated technological progress and improved living standards, they have simultaneously introduced one of the most pressing environmental problems of the 21st century: the sustainable disposal and recycling of heterogeneous, polymer-rich waste streams.

Plastics alone have surpassed an annual production of 400 million metric tons, a figure expected to triple by 2050 if current trends continue. A significant portion of this waste arises from single-use products, including personal protective equipment (PPEs), the latter of which saw explosive growth during the COVID-19 pandemic. Composite waste, such as decommissioned wind turbine blades (WTBs) and end-of-life photovoltaic (PV) panels, adds another layer of complexity due to their combination of polymers, glass fibers, and metals. Municipal solid waste (MSW), already exceeding 2 billion tons annually, further contributes to this problem as it contains a diverse mixture of plastics, biomass, textiles, and inorganics that are often not effectively separated or treated. Collectively, these waste streams represent a massive environmental burden, as they are either landfilled, incinerated, or inadequately recycled, leading to the loss of valuable resources, the generation of greenhouse gases, and the accumulation of persistent pollutants in ecosystems.

Recognizing the magnitude of these challenges, the European Union (EU) has positioned itself as a leader in advancing sustainable waste management policies. The Waste Framework Directive (2008/98/EC) established the "waste hierarchy," prioritizing prevention, reuse, recycling, and energy recovery before final disposal. Complementary to this, the Landfill Directive set ambitious targets to reduce biodegradable waste sent to landfills, aiming to curb methane emissions and environmental degradation. More recently, the European Green Deal and the Circular Economy Action Plan 2.0 have broadened this vision by decoupling economic growth from resource consumption, promoting systemic changes across industries, and supporting innovative technologies for waste valorization. In the energy sector, Renewable Energy Directives (REDII and REDIII) highlight the importance of sustainable bioenergy, increased recycling, and binding targets for renewable energy consumption. Together, these policy frameworks provide a strong justification for exploring advanced thermochemical recycling approaches that not only divert waste from landfills but also produce valuable resources.

Despite the existence of these frameworks, practical waste management remains plagued by technological and economic limitations. Mechanical recycling, while widely practiced, is only applicable to certain clean and sorted thermoplastics and often results in "downcycling," where the quality of recycled material is inferior to the original. Incineration, though effective in reducing waste volume and generating energy, releases hazardous emissions such as dioxins, furans, and CO<sub>2</sub>, making it environmentally controversial and increasingly restricted under EU climate targets. Pyrolysis, another thermal route, converts plastics into liquid fuels but requires

high temperatures (400–800 °C) and significant energy input, raising questions about economic feasibility and environmental performance. Consequently, these conventional methods are insufficient to address the rising volumes of complex waste streams, especially composites like WTBs and PV panels that contain both organic and inorganic fractions.

It is within this context that the current doctoral research introduces oxidative liquefaction as a sustainable alternative. This process operates under high-temperature, high-pressure conditions with an oxidizing agent to selectively degrade the polymeric matrix of waste materials. Unlike pyrolysis, oxidative liquefaction operates at lower energy requirements and facilitates the production of oxygenated chemical compounds (OCCs), volatile fatty acids, and other secondary products. Importantly, it also enables material recovery, such as the extraction of clean glass fibers from WTBs, which would otherwise be lost in incineration or landfill disposal. By addressing both energy efficiency and material recovery, oxidative liquefaction aligns strongly with circular economy principles and EU policy goals.

The chapter also identifies several critical research gaps that this thesis aims to address. First, there is limited knowledge about the effects of different reaction parameters—temperature, pressure, oxidant concentration, reaction time, and waste-to-liquid ratio—on process efficiency and product distribution. Second, existing research has rarely applied oxidative liquefaction to highly complex waste streams such as WTBs, PV panels, or mixed MSW, which behave differently compared to homogeneous plastic waste. Third, there is a lack of comparative studies assessing the adaptability of the process across different feedstocks, making it difficult to design universal strategies. Fourth, optimization of the process for higher yields and reduced energy input is still in its infancy. Finally, most reported work remains confined to laboratory scale, with significant challenges in scaling up due to reactor design, process economics, and regulatory acceptance.

To bridge these gaps, the doctoral study sets out a clear aim: to develop and optimize oxidative liquefaction as an advanced chemical recycling technique for diverse, polymer-containing waste streams. The objective is to maximize polymer degradation, enhance the production of valuable OCCs, and recover secondary raw materials, all while improving energy efficiency. By doing so, the research not only contributes to scientific understanding of waste conversion processes but also provides actionable knowledge for future industrial applications

## 2 Materials and Methods

The second chapter of this thesis describes in detail the materials investigated, the characterization methods employed, and the experimental framework developed to assess oxidative liquefaction as a recycling pathway for complex waste streams. Since the core objective of this study is to evaluate the potential of oxidative liquefaction across diverse waste types, it was essential to adopt a systematic and scientifically rigorous methodology that ensured comparability, reproducibility, and meaningful interpretation of results.

#### 2.1 Selection of Waste Materials

Four distinct categories of waste were chosen, each representing a critical and emerging environmental challenge. End-of-life WTBs were selected as a prime example of composite waste arising from the renewable energy sector. These blades, constructed primarily from fiber-reinforced polymers (FRPs), are difficult to recycle using conventional methods due to their combination of glass fibers, polymeric resins, and fillers.

The second category was PPE, which has contributed significantly to plastic pollution, particularly during and after the COVID-19 pandemic. PPEs, such as masks, gloves, gowns, and respirators, are predominantly composed of polypropylene (PP), polyethylene (PE), and polyester (PES). Their relatively homogeneous polymeric composition makes them an interesting counterpart to heterogeneous waste streams.

The third material type, end-of-life PV panels, reflects the growing challenge posed by decommissioned renewable energy infrastructure. PV panels typically consist of glass, silicon-based layers, encapsulant polymers (e.g., ethylene-vinyl acetate), and trace metals. Their complex, layered structure makes them particularly difficult to recycle without advanced processes.

Finally, MSW was included to represent one of the most widespread and heterogeneous waste streams globally. MSW typically consists of plastics, textiles, organics, paper, metals, and glass. Its inclusion ensured that oxidative liquefaction was tested under highly realistic and complex conditions, thereby assessing its adaptability for real-world scenarios.

#### 2.2 Feedstock Characterization

To understand the intrinsic properties of each waste stream, baseline proximate and ultimate analyses were carried out. Proximate analysis provided information on moisture, ash, and volatile matter content, while ultimate analysis quantified elemental composition, including carbon, hydrogen, nitrogen, sulfur, and oxygen. These results were critical for determining the energy potential, degradation behavior, and chemical recovery prospects of each feedstock.

Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify functional groups within the polymeric structures of the materials. This technique revealed chemical bonds susceptible to oxidative attack, providing insight into possible degradation pathways. Scanning Electron Microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS) offered visual

evidence of material morphology and surface changes, while also confirming elemental composition in solid residues.

To further analyze thermal behavior, Thermogravimetric Analysis (TGA) was conducted. TGA profiles demonstrated the degradation patterns of different materials, revealing whether they decomposed in single or multiple stages. For example, PPEs showed uniform, single-step degradation, while WTBs and PV panels displayed multi-stage processes due to their composite nature. These results informed subsequent reactor operation parameters.

#### 2.3 Reactor Design and Experimental Framework

The experimental core of this thesis was the development of a custom-built, high-temperature, high-pressure batch reactor capable of handling the oxidative liquefaction process. The reactor was designed to withstand pressures up to several MPa and temperatures in the range of 250–400 °C, creating an environment conducive to polymer breakdown in the presence of oxidants. Key design features included a robust stainless steel chamber, precise temperature control, and monitoring instruments to record internal temperature profiles during each run.

Waste samples, after being cut and homogenized into defined particle sizes, were loaded into the reactor with a defined ratio of oxidant and solvent medium. The waste-to-liquid ratio (W/L), oxidant concentration, temperature, pressure, and residence time were systematically varied to study their effect on degradation efficiency and product yields.

#### 2.4 Experimental Design and Statistical Methods

Given the multi-variable nature of the process, a statistically robust experimental design was crucial. A Response Surface Methodology (RSM) approach was adopted to explore interactions between variables and identify optimal process conditions. The experimental matrix was generated using a central composite face-centered design, enabling efficient investigation of nonlinear effects and parameter interactions with a limited number of experiments.

In addition, Analysis of Variance (ANOVA) was employed to statistically evaluate the significance of individual factors and their interactions. This approach ensured that observed differences in outcomes, such as total solid reduction (TSR) or OCCs yields, were attributable to process variables rather than experimental noise.

## 2.5 Product Analysis

After each experimental run, the products were separated into solid and liquid fractions. TSR and Total Polymer Degradation (TPD) were calculated as indicators of how effectively the polymeric matrix had been broken down. The liquid products were subjected to Gas Chromatography with Flame Ionization Detection (GC-FID), a technique capable of quantifying individual oxygenated compounds, volatile fatty acids, and other secondary chemicals. This step was critical for evaluating the economic and industrial potential of oxidative liquefaction, since the production of valuable OCCs is central to its viability as a recycling technology.

# 3 Oxidative Liquefaction of Wind Turbine Blades and Product Analysis

WTBs represent one of the fastest-growing categories of composite waste. As global wind power capacity expands, the decommissioning of turbines has created an urgent waste management challenge, with millions of tonnes of blade material projected to enter the waste stream in coming decades. These blades, primarily composed of fiber-reinforced polymers (FRPs), combine high-strength glass fibers with thermosetting polymeric resins. Their durability is advantageous in service but problematic at end-of-life, as cross-linked resins resist melting and reprocessing. Conventional disposal routes such as landfilling, incineration, and mechanical shredding are environmentally damaging or yield only low-value outputs. Against this backdrop, oxidative liquefaction is proposed in this thesis as an innovative, thermochemical method that enables both resin degradation and fiber recovery while also producing valuable chemical compounds.

Chapters 3 and 4 together document the experimental application of oxidative liquefaction to WTBs and provide a thorough characterization of the resulting products. Chapter 3 focuses on degradation behavior, fiber recovery, and process optimization, while Chapter 4 offers an indepth analysis of the liquid and solid fractions to evaluate their potential for industrial reuse.

## 3.1 Experimental Investigation of Oxidative Liquefaction of WTBs

The initial investigation involved preparing WTB samples into uniform chips, which were then treated in a high-pressure, high-temperature batch reactor developed for this project. The process operated at temperatures between 250–350 °C and under several MPa of pressure, with oxidants introduced to promote selective degradation of the resin matrix. Key operational parameters—temperature, pressure, oxidant concentration, residence time, and waste-to-liquid ratio (W/L)—were systematically varied using a statistical design of experiments approach.

The central performance indicators included TSR and TPD, which measured the extent of resin breakdown, alongside the yield of OCCs in the liquid fraction. By applying Response Surface Methodology (RSM) and Analysis of Variance (ANOVA), the study optimized conditions to maximize degradation efficiency and product recovery.

#### 3.1.1 Resin Degradation and Fiber Recovery

Results showed that oxidative liquefaction effectively degraded the epoxy and polyester resin matrix, exposing clean, undamaged glass fibers. SEM the removal of resin residues, revealing surfaces comparable to virgin fibers. This outcome is critical because conventional methods such as pyrolysis often damage fibers or leave them contaminated with char, limiting reuse. Here, the oxidative environment not only facilitated chemical conversion but also preserved fiber integrity, positioning the process as a dual waste-to-resource pathway.

TSR values increased significantly at higher temperatures and moderate oxidant levels, confirming the thermal-oxidative sensitivity of resin polymers. However, excessive oxidant concentrations caused partial etching of glass fibers, highlighting the need for precise optimization.

#### 3.1.2 Energy Consumption and Efficiency

A common critique of thermochemical recycling is its high energy demand. To address this, energy consumption was carefully monitored. Comparative analysis indicated that oxidative liquefaction requires substantially less energy than pyrolysis (which typically operates above 400 °C). Inverse heating analysis revealed distinct reaction stages: initial polymer softening, chain scission, and full oxidative breakdown. Identifying these stages provided insights into how energy input could be minimized without sacrificing resin degradation. Overall, the process demonstrated a favorable energy-to-yield ratio, suggesting industrial scalability.

## 3.2 Product Analysis from Oxidative Liquefaction of WTBs

While Chapter 3 established the feasibility of degradation and fiber recovery, Chapter 4 extends the investigation by analyzing the chemical and structural composition of the products. Understanding product quality is essential to assess whether oxidative liquefaction can deliver materials and chemicals of commercial value.

#### 3.2.1 Solid Fraction: Fiber Quality

The recovered glass fibers were examined using SEM-EDS, which confirmed near-complete removal of resin residues. Energy-dispersive spectroscopy (EDS) revealed the presence of silicon, calcium, and aluminum typical of clean glass fibers, with negligible organic carbon contamination. This suggests that the fibers could be reintroduced into composite manufacturing, closing the loop on material cycles. The economic value of recovered fibers—although lower than virgin glass fibers—represents a significant step toward circularity in the wind energy sector.

#### 3.2.2 Liquid Fraction: Oxygenated Chemical Compounds

The liquid phase was analyzed using GC-FID. Results showed a rich distribution of oxygenated compounds, including volatile fatty acids (VFAs), aldehydes, ketones, and phenolic derivatives. VFAs such as acetic acid and propionic acid were among the most prominent products, indicating potential for use in chemical and biochemical industries.

The yield and composition of OCCs were highly sensitive to process conditions. Higher temperatures promoted full polymer breakdown, yielding low-molecular-weight VFAs, while lower temperatures favored partially oxidized compounds. This tunability demonstrates that oxidative liquefaction can be adjusted depending on desired outputs—whether maximizing resin degradation, fiber recovery, or liquid chemicals.

#### 3.2.3 FTIR Structural Insights

FTIR further confirmed chemical changes in the polymer matrix. Key absorption peaks corresponding to C=O, C-O, and O-H functional groups increased after treatment, consistent with the formation of oxygenated degradation products. The reduction in C-H and aromatic peaks indicated resin depolymerization. These observations corroborated GC-FID findings and validated oxidative liquefaction as a selective, oxidation-driven process.

#### 3.2.4 Optimization of Product Yields

By combining the structural, chemical, and statistical analyses, the research identified optimal process windows that balance multiple outcomes: (i) maximum TSR, (ii) high OCC and VFA yields, and (iii) minimal energy consumption. Importantly, the optimization revealed trade-offs—for example, higher TSR coincided with higher VFAs but sometimes reduced fiber quality due to over-oxidation. Thus, operational flexibility is key for tailoring the process to specific industrial goals.

## 3.3 Combined Outcomes and Implications

Together, Chapters 3 and 4 provide compelling evidence that oxidative liquefaction is a comprehensive recycling solution for WTBs. Unlike traditional methods, it offers simultaneous:

- 1. **Resin degradation** breaking down thermosetting polymers that are otherwise non-recyclable.
- 2. **Fiber recovery** delivering clean, reusable glass fibers suitable for reintroduction into composite manufacturing.
- 3. Chemical production generating oxygenated compounds of industrial relevance, including VFAs, which have applications in bioplastics, food additives, and green solvents.
- 4. **Energy efficiency** achieving significant outputs at lower energy inputs than conventional waste management techniques.

These findings align closely with EU policy objectives for waste reduction, resource valorization, and circular economy advancement. They also demonstrate the **scalability potential** of the technology, although challenges remain in reactor design, continuous operation, and downstream purification of liquid product

# 4 Comparative Study – Oxidative Liquefaction of PPE Waste and WTBs

This chapter provides a comparative evaluation of oxidative liquefaction applied to PPE waste and WTBs, two distinct yet environmentally significant waste streams. The purpose of this analysis was to examine how the intrinsic differences in feedstock composition influence degradation behavior, product yields, and recovery potential, and thereby assess the flexibility of oxidative liquefaction as a universal recycling strategy.

#### 4.1 Feedstock Characteristics and Thermal Behavior

TGA was employed as a diagnostic tool to investigate degradation pathways. PPE waste, which primarily consists of polypropylene (PP), polyethylene (PE), and polyester (PES), displayed a single-stage degradation profile. This behavior is consistent with the relatively homogeneous and thermoplastic nature of PPEs, where polymer chains undergo cleavage within a narrow temperature range, leading to complete volatilization of the organic fraction.

In contrast, WTBs—composed of fiber-reinforced polymers (FRPs)—exhibited multi-stage degradation. The initial stages corresponded to the decomposition of polymeric resins, followed by progressive breakdown of fillers and partial degradation of surface coatings. A final stage associated with inorganic residue stability was observed, reflecting the heterogeneous structure of the composite. These differences underscore the importance of tailoring process parameters to the chemical and structural characteristics of each waste type.

## 4.2 Resin Degradation and Fiber Recovery

The oxidative liquefaction of WTBs resulted in effective resin removal and recovery of clean glass fibers, as confirmed by SEM-EDS analysis. Fiber recovery is one of the most valuable outcomes of treating FRPs, since it provides secondary raw materials for new composite manufacturing. By contrast, PPE waste did not contain reinforcing fibers, and thus the solid residue after treatment was minimal and primarily composed of ash. While this meant that no material recovery pathway was available for PPEs, the absence of inorganic reinforcement simplified degradation and contributed to higher chemical yields.

## 4.3 Liquid Product Distribution and OCC Yields

GC-FID revealed marked differences in the chemical composition of the liquid fractions. PPE waste generated higher yields of OCCs, particularly VFAs such as acetic and propionic acids, along with ketones and aldehydes. This outcome is linked to the uniform polymeric composition of PPEs, which facilitated more complete breakdown into low-molecular-weight oxygenated products.

In contrast, WTBs produced a more diverse mixture of products. While OCCs were still present in significant amounts, the yields were comparatively lower than those from PPE waste. The presence of fillers and glass fibers likely diverted some of the oxidant, reducing the overall efficiency of resin-to-OCC conversion. Nonetheless, the recovery of valuable solid fibers

alongside moderate chemical yields represents a balanced outcome, with both material and chemical valorization achieved simultaneously.

## 4.4 Flexibility and Process Optimization

The comparative study demonstrates the adaptability of oxidative liquefaction across very different waste types. For PPEs, the method excels in maximizing chemical recovery, converting nearly the entire organic fraction into useful oxygenated compounds. For WTBs, it provides dual benefits—chemical recovery and fiber reclamation—although optimization is required to balance these outcomes. Statistical analysis using RSM and ANOVA confirmed that while temperature and oxidant concentration were critical factors for both waste types, the optimal values differed significantly between PPEs and WTBs.

## Chapter 6

# 5 Municipal Solid Waste Generation

This chapter addresses the growing issue of MSW, one of the most widespread and challenging waste streams worldwide. MSW refers to household and urban waste, typically composed of plastics, textiles, food residues, paper, cardboard, metals, and glass. According to World Bank estimates, global MSW generation exceeds 2 billion tonnes annually, with projections suggesting a further increase to 3.4 billion tonnes by 2050 if current trends persist. This rapid growth is driven by rising urbanization, population expansion, and changing consumption patterns, making MSW management a central concern for both developed and developing nations.

The composition of MSW varies geographically but is generally characterized by a high fraction of organic matter, significant amounts of plastics, and heterogeneous mixtures that complicate recycling. In the European Union, and particularly in Poland, waste separation at the source has improved in recent years due to regulatory frameworks and public awareness campaigns. Nonetheless, large fractions of MSW remain non-recyclable or are contaminated to the extent that they cannot be processed through conventional mechanical or biological methods. Plastics, multilayer packaging, and composite items are especially problematic because they resist biodegradation and often evade existing recycling infrastructure.

Current MSW management strategies include mechanical recycling, composting of biodegradable fractions, incineration with energy recovery, and landfilling. While incineration reduces waste volume and generates electricity or heat, it is energy-intensive and produces harmful emissions. Landfilling, although still widely practiced, leads to long-term environmental risks such as leachate contamination and methane emissions. Mechanical recycling, though effective for certain plastics and paper fractions, is limited by contamination and material downcycling.

Given these shortcomings, the chapter positions oxidative liquefaction as a potential alternative pathway for treating unrecyclable MSW fractions. By converting heterogeneous waste into valuable oxygenated chemicals while reducing solid residues, this technology aligns with EU circular economy goals and offers a promising route for addressing the limitations of current MSW treatment approaches.

## 6 Oxidative Liquefaction of MSW vs PPEs

This chapter presents the experimental application of oxidative liquefaction to MSW and provides a comparative analysis with results previously obtained for PPEs waste. The aim of this comparison was to test the adaptability of the process across two distinct categories of feedstock—heterogeneous MSW with its complex mixture of organic and inorganic fractions, and homogeneous PPEs, which are predominantly composed of single-type polymers. The findings underscore both the challenges and opportunities of extending oxidative liquefaction beyond uniform waste streams to real-world, highly variable MSW.

#### 6.1 Characteristics of MSW vs PPEs

Baseline analyses revealed clear differences between the two feedstocks. MSW displayed high ash content due to the presence of glass, metals, dirt, and inert materials. Its proximate and ultimate analysis also confirmed significant heterogeneity, with variable fractions of plastics, biomass, and paper. In contrast, PPEs showed low ash content and a consistent polymeric composition dominated by polypropylene (PP) and polyethylene (PE). These differences strongly influenced degradation patterns and product yields.

TGA confirmed that PPEs degraded in a single, sharp thermal stage, consistent with the uniformity of their polymer structure. MSW, on the other hand, underwent multi-stage degradation, reflecting its diverse composition. Initial stages corresponded to the breakdown of organics and polymers, while later stages involved slower decomposition of lignocellulosic fractions and mineral-rich residues. This multi-stage profile necessitated careful adjustment of process conditions to ensure effective treatment.

## **6.2 Experimental Outcomes**

When subjected to oxidative liquefaction, MSW demonstrated lower TSR compared to PPEs. The high ash fraction limited the extent of organic degradation, as non-combustible materials diluted the feedstock. Nonetheless, significant polymer breakdown was achieved, and the process successfully converted complex fractions into a liquid phase rich in oxygenated compounds.

GC-FID analysis revealed that PPEs produced higher overall OCC yields, particularly VFAs, owing to their simpler composition. MSW produced a more diverse product spectrum, including VFAs, phenolic compounds, and partially oxidized intermediates. While yields per unit of waste were lower than for PPEs, the results demonstrated that even highly mixed waste can be valorized into useful chemical streams.

## 6.3 Energy Efficiency and Process Optimization

Energy consumption analysis showed that MSW treatment was less efficient than PPE liquefaction, largely due to the energy required to overcome its heterogeneity and mineral content. However, optimization using Response Surface Methodology (RSM) and Analysis of Variance (ANOVA) identified operational windows that significantly improved TSR and OCC yields. For MSW, slightly higher temperatures and longer residence times proved necessary

compared to PPEs, reflecting the more complex degradation profile. Oxidant concentration also emerged as a critical factor, requiring careful balance to avoid excessive oxidation of already mineral-rich residues.

## 6.4 Adaptability of Oxidative Liquefaction

The comparative analysis illustrates the flexibility of oxidative liquefaction as a treatment method. For PPEs, the process excels in maximizing chemical recovery from polymer-rich, homogeneous waste. For MSW, while challenges such as lower TSR and reduced efficiency are evident, oxidative liquefaction nonetheless demonstrated the ability to convert heterogeneous waste into valuable oxygenated compounds. This adaptability is particularly important given the increasing urgency to manage MSW sustainably.

# 7 Oxidative Liquefaction of Photovoltaic Panels

This chapter explores the application of oxidative liquefaction to end-of-life PV panels, an emerging waste stream of growing global concern. With the rapid expansion of solar energy, millions of PV modules are expected to reach the end of their service life over the next two decades. Estimates suggest that by 2050, cumulative PV waste could exceed 60–70 million tonnes worldwide. PV panels are complex, multi-layered structures typically consisting of glass (70–75%), polymers such as ethylene-vinyl acetate (EVA), silicon-based cells, aluminum frames, and trace amounts of metals including silver and tin. Their heterogeneous composition and high ash content make them difficult to recycle using conventional methods. Mechanical processes can recover bulk glass and metals, but they fail to effectively treat polymeric encapsulants, which represent a significant fraction of the waste. Oxidative liquefaction is therefore investigated here as a potential pathway for valorizing the organic portion of PV panels while simultaneously reducing waste volume.

#### 7.1 Material Characterization and Degradation Behavior

Comprehensive characterization of PV waste confirmed its heterogeneous nature, with a high inorganic fraction dominated by glass and metals, alongside polymer encapsulants and backsheet materials. TGA revealed a multi-stage degradation profile, with distinct phases corresponding to the breakdown of polymer layers, slow degradation of additives, and persistence of mineral-rich residues. Compared with PPEs or homogeneous plastics, the thermal stability of PV panels was higher, reflecting the protective role of glass and metallic components.

SEM-EDS analysis of treated residues further demonstrated structural and compositional changes. Post-treatment samples showed removal of polymeric coatings and surface oxidation of glass particles, while EDS spectra confirmed reductions in organic carbon peaks. These results provided clear evidence that oxidative liquefaction successfully degraded the polymer fraction embedded within the layered PV structure.

## 7.2 Liquid Product Formation

Despite the high ash content of PV waste, oxidative liquefaction produced valuable liquid fractions rich in OCCs. GC-FID analysis identified a mixture of VFAs, aldehydes, and partially oxidized intermediates. Although yields were lower on a per-mass basis compared to PPEs or WTBs, the production of VFAs such as acetic acid demonstrated that even complex PV waste can generate chemicals of industrial interest. The diversity of liquid products also indicates potential opportunities for downstream separation and upgrading into higher-value compounds.

## 7.3 Optimization of Process Conditions

Process optimization was performed using Response Surface Methodology (RSM) to evaluate the effects of temperature, oxidant concentration, and residence time. Results indicated that slightly higher operating temperatures and longer residence times were required compared to PPEs to achieve significant polymer breakdown in PV waste. Optimal conditions balanced the degradation of encapsulant polymers with the avoidance of excessive energy consumption. Although TSR values were lower than for other waste streams due to the high inorganic content, the process still achieved meaningful reductions in the polymeric fraction and conversion into OCCs.

#### Feasibility and Challenges

The findings confirm the technical feasibility of applying oxidative liquefaction to PV panels. The process effectively addresses the organic fraction of PV waste, offering a complementary pathway alongside mechanical recycling of glass and metals. However, several challenges remain. First, the high ash fraction limits overall TSR and reduces chemical yields, which may impact economic viability. Second, scaling the process to industrial levels will require reactor designs capable of handling heterogeneous, abrasive, and mineral-rich feedstocks. Third, further work is needed to improve downstream separation and purification of the liquid products to maximize their commercial value.

## **Summary and Conclusions of the Study**

This doctoral study systematically investigated oxidative liquefaction as an advanced recycling strategy for diverse polymer-rich waste streams, including WTBs, PPE, MSW, and PV panels. The research demonstrated that oxidative liquefaction can simultaneously achieve polymer degradation, material recovery, and production of oxygenated chemical compounds OCCs, offering a versatile and energy-efficient alternative to conventional mechanical or thermal recycling methods.

#### Key findings include:

#### 1. Effectiveness Across Feedstocks:

- WTBs, composed of fiber-reinforced polymers, underwent multi-stage resin degradation, enabling recovery of clean glass fibers suitable for reuse in composites.
- o PPE waste, largely homogeneous polymers, showed single-stage degradation and produced high yields of OCCs, particularly volatile fatty acids.
- MSW, characterized by heterogeneity and high ash content, required feedstockspecific optimization but was successfully converted into liquid OCCs, demonstrating process adaptability.
- PV panels, despite their complex, yielded significant polymer breakdown and production of OCCs, highlighting the method's applicability to multi-layered waste.

#### 2. Product Characterization and Valorization:

- SEM-EDS and FTIR analyses confirmed selective polymer degradation and preservation of valuable solid materials.
- o GC-FID analysis identified a range of OCCs, including VFAs, aldehydes, ketones, and phenolic compounds, which hold potential as chemical feedstocks.
- o Optimal process parameters were feedstock-dependent, with temperature, oxidant concentration, and residence time being critical factors.

#### 3. Energy Efficiency and Process Optimization:

- Oxidative liquefaction operated at lower temperatures than pyrolysis (250–400 °C), reducing energy consumption.
- Response Surface Methodology (RSM) and ANOVA enabled identification of operational windows balancing total solid reduction, OCC yield, fiber recovery, and energy efficiency.

#### 4. Flexibility and Circular Economy Implications:

o The process accommodates both homogeneous and heterogeneous waste streams, providing a flexible platform for industrial implementation.

 Recovery of fibers from WTBs and production of chemically valuable liquids align with circular economy principles and EU policy goals for sustainable waste management.

#### **Conclusions:**

The study concludes that oxidative liquefaction is a robust and adaptable technology for recycling polymer-rich wastes. It successfully addresses the limitations of conventional mechanical, incineration, and pyrolysis methods by offering dual benefits of material recovery and chemical valorization while maintaining energy efficiency. Feedstock-specific optimization is essential to maximize outcomes, but the method's versatility positions it as a promising solution for industrial-scale application. Future work should focus on reactor design for continuous operation, scaling-up, and downstream processing to enhance product purity and economic feasibility.

Overall, this research provides a comprehensive foundation for the development of advanced chemical recycling strategies, supporting the transition toward a circular, resource-efficient, and environmentally sustainable waste management framework.