



National University of Science and Technology
"POLITEHNICA" Bucharest
Faculty of Power Engineering

Doctoral School of Energy Engineering
060042 București, Splaiul Independenței, nr. 313, sector 6



SUMMARY

DOCTORAL THESIS

STUDY ON THE OPTIMIZATION OF THE PYROLYSIS
PROCESS FOR ENERGY RECOVERY FROM POLYMER
WASTE

Author: PhD Candidate Eng. Mircea Gabriel MACAVEI

PhD Supervisor: Prof. Dr. Eng. Cosmin MĂRCULESCU

Bucharest, 2025

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1. INTRODUCTION AND GENERAL CONTEXT OF THE RESEARCH

1.1 STATEMENT

Polymer waste has become a major global challenge due to the ever-increasing volume of plastics produced and deficiencies in their waste management. Although recent years have seen a strong emphasis on developing collection and recycling systems, the majority of plastic waste is still not efficiently recovered – most ends up in landfills or is disposed of uncontrolled, reaching natural ecosystems [1].

One promising router for plastic waste recovery is thermochemical recycling via pyrolysis, a process of thermal degradation of polymers in the absence of oxygen. Through pyrolysis, polymer macromolecules are cracked into smaller molecules, yielding three main categories of by-products: pyrolysis gas (combustible, rich in compounds such as H_2 , CH_4 and light hydrocarbons), oil (a liquid fraction composed of medium-molecular-weight hydrocarbons that can be used as a liquid fuel), and a solid called char. Unlike incineration, which involves complete oxidation of waste, pyrolysis takes place in a controlled, non-oxidizing environment, leading to reduced pollutant emissions and allowing recovery of a significant portion of the waste's chemical energy in the form of useful products. For example, pyrolysis oil obtained from plastic has a calorific value comparable to conventional fuels and can be used as a substitute after refining [2]. Thus, pyrolysis offers a chemical recycling pathway that contributes to the circular economy and closes the waste-to-resource loop.

In this context, microwave-assisted pyrolysis has emerged as an alternative aimed at overcoming some of the limitations of conventional heating. The basic principle is the use of microwave energy to directly heat the waste material from within at the molecular level, through the interaction of the electromagnetic field with the material or with an added susceptor agent [3].

Unlike the conventional heating mechanism, which heats the sample from the surface inward, microwaves penetrate the material's volume and can generate heat at the molecular level from within the sample. This volumetric heating mechanism leads to much faster and more uniform heating [4], potentially reducing the time needed to reach pyrolysis temperature and resulting in energy savings compared to conductive heating. Moreover, microwave processing can increase the conversion yield into useful products and improve their quality [5].

1.2 AIM AND OBJECTIVES OF THE RESEARCH

The general aim of the research is to optimize the pyrolysis process for the energy recovery of polymer waste, with a focus on conventional and microwave-assisted pyrolysis technology applied to low-density polyethylene (LDPE). To achieve this aim, the following specific research objectives were established:

- Characterization of the LDPE polymer waste used as raw material, in terms of chemical composition (elemental analysis for C, H, N, S, with oxygen content estimated), relevant physical properties (density, molecular structure via FTIR spectroscopy, degree of crystallinity), and energy potential (volatile matter content, ash content, and calorific value). The initial characterization aims to highlight to what extent the LDPE waste is suitable for recovery through pyrolysis (for example, a high carbon and hydrogen content and negligible ash content indicate a high potential for conversion into volatile products).

- Study of the thermal behavior of LDPE via thermogravimetric analyses (TGA), in order to determine characteristic thermal parameters (onset temperature of degradation and devolatilization intervals, mass loss as a function of temperature).
- Determination of the reaction kinetics of the LDPE pyrolysis process, based on the obtained experimental data. This objective involves identifying the kinetic models and parameters (activation energies, pre-exponential factors) that best describe the polymer's thermal decomposition, by applying various kinetic methods (isoconversional methods and modeling based on predefined reaction mechanisms)
- Optimization of the pyrolysis process under conventional conditions. Based on information from the literature, experimental campaigns of conventional pyrolysis of LDPE waste were carried out, seeking to optimize the process by introducing high-performance catalysts and co-pyrolysis materials.
- **Identification and selection of suitable microwave susceptor materials for LDPE pyrolysis.** Given that LDPE is a poor absorber of microwave energy, introducing a susceptor (a material with high dielectric losses that heats efficiently under microwave fields) is necessary to initiate and sustain the process. Two types of susceptors were investigated (biochar derived from biomass and graphite), evaluating their performance in absorbing microwaves and converting them into thermal energy. The objective is to select the optimal susceptor that ensures rapid and uniform heating of the LDPE waste.
- **Optimization of process parameters for microwave-assisted pyrolysis,** based on laboratory experiments. The goal was to determine optimal conditions that maximize LDPE conversion and the yields of desired products. For example, adjusting the microwave power and susceptor position can significantly influence the proportion of liquid product versus gaseous product, as well as the process's energy consumption. The optimization aimed both to achieve as high an energy efficiency as possible and to control the product fraction distribution toward maximizing the most valuable fraction from an energy standpoint.
- **Synthesis of bio-catalysts based on biochar and other supports,** used with the purpose of improving the pyrolysis process. In this study, new catalysts were synthesized by impregnating metals (Ni, Fe) onto supports of activated biochar or other materials (ash, zeolites), using microwave- and ultrasound-assisted methods to achieve uniform precursor dispersion. The purpose of these catalysts is twofold: (i) to act as susceptors within the LDPE waste mass and (ii) to catalyze cracking reactions. These catalysts were studied, tested, and characterized, and their influence on the distribution of pyrolysis products was assessed.
- **Analysis of the distribution of products obtained by pyrolysis (gas, liquid, solid) and evaluation of their quality.** Following the experiments, the quantities of each fraction produced were determined, and their composition was characterized. For the gas fraction, the concentrations of main components (H₂, CH₄, CO, CO₂, light hydrocarbons, etc.) were measured using chromatographic analysis, highlighting how different catalysts or process conditions alter the gas composition. Additionally, for the liquid, wax, and gas fractions produced, the energy potential (calorific value) was determined.

1.3 THESIS STRUCTURE

The thesis is organized into seven chapters, as follows:

Chapter 1 – Introduction and general context of the research. This chapter presents the issues underlying the work (polymer waste management and the need for their energy recovery), and a comparative analysis of current technologies for managing polymer waste, with emphasis on conventional pyrolysis and microwave-assisted pyrolysis.

Chapter 2 – Types of polymer waste and pyrolysis processes for recovery. In this chapter, the literature on methods for recycling and recovering plastic waste is reviewed. Conventional methods (mechanical recycling, energy recovery through incineration) and their limitations are discussed. Then, thermochemical conversion processes, especially pyrolysis, are detailed. The evolution of plastic pyrolysis technologies is outlined, from classical processes to microwave-assisted ones, highlighting the advantages and disadvantages of each.

Chapter 3 – Physico-chemical characterization of LDPE waste. This chapter is dedicated to describing the properties of the polymer waste used in the experimental investigations. The first part of the chapter provides a detailed characterization of the LDPE waste (elemental and proximate chemical composition, structural analysis via FTIR, and determination of calorific value). Then, the thermogravimetric studies (TGA/DTG) performed on LDPE samples are presented, both under gradual heating and direct exposure to set temperatures. The observed degradation kinetics are presented and the kinetic parameters of the LDPE pyrolysis process are identified.

Chapter 4 – Experimental studies on the conventional pyrolysis of LDPE. In this chapter, the potential for energy recovery of LDPE waste under conventional conditions was studied by conducting three types of experimental campaigns: (i) conventional pyrolysis at various process temperatures and heating rates, (ii) optimization of these processes using catalysts, and (iii) co-pyrolysis experiments of LDPE with a biomass material in the form of chicken bone waste. By analyzing the mass balance and pyrolysis gas composition, the effect of these experimental configurations on the LDPE recovery process was investigated.

Chapter 5 – Experimental studies on microwave-assisted pyrolysis of LDPE. This chapter focuses on investigating how microwaves can be efficiently used to convert LDPE waste into useful products, aiming to improve and optimize the process. The first part presents results regarding the choice of the optimal susceptor material. Several susceptor materials were tested (such as biochar derived from biomass and standard materials like graphite), evaluating their microwave absorption capacity. Next, the chapter details microwave-assisted pyrolysis experiments conducted at different powers and conditions. Two main microwave configurations were investigated: (i) a 2.45 GHz system with 200 W power, and (ii) a 915 MHz system with 400 W power. Chapter 5 also includes investigations on activating the biochar resulted from pyrolysis, as well as the synthesis of biochar- and ash-based catalysts using a combined microwave-ultrasound (MW-US) methodology.

Chapter 6 – Energy balance of the conversion processes used. This chapter presents the energy analysis of the studied processes. An important aspect addressed in Chapter 6 is the energy balance of the pyrolysis process, comparing the two studied processes: conventional pyrolysis and microwave-assisted pyrolysis. Based on experimental data and thermodynamic calculations, the energy contributions required for the studied conversion processes were determined, as well as for each transformation stage within these processes.

Chapter 7 – General conclusions and original contributions. The work concludes with a synthesis of the main scientific and technical findings obtained during the research. It summarizes how the proposed objectives were achieved and highlights the original contributions of the thesis in the field of energy recovery of polymer waste through conventional and microwave-assisted pyrolysis.

2. TYPES OF POLYMER WASTE AND PYROLYSIS PROCESSES FOR VALORIZATION

2.1 FUNDAMENTAL CHARACTERISTICS OF POLYMERS AND PLASTICS

Plastics are, in particular, synthetic organic polymers defined by their characteristic plasticity (the ability to permanently deform under an external force without cracking or breaking), owing to the covalent bonds in their long chains of monomers.

Thermoplastic polymers represent the broadest class of plastics. Thermoplastics can be melted by heating and solidified by cooling repeatedly, allowing easy processing [6]. Polyolefins fall into this category. These are polymers composed exclusively of carbon and hydrogen, obtained by polymerizing alkenes (hydrocarbons with a C=C double bond). The main polymers are polyethylene (PE) and polypropylene (PP), which also have the largest share in total plastic production and consumption, due to their ease of production, useful properties, and relatively low cost [7].

PE is obtained by polymerizing the monomer ethylene under various pressures. The main types of PE are: low-density polyethylene (LDPE), high-density polyethylene (HDPE), and medium-density polyethylene (MDPE). LDPE is produced under high pressure, whereas HDPE is produced under low pressure [8]. As the degree of branching increases, their density decreases. PE is used predominantly for packaging (films, bags, bottles), consumer goods, toys, pipes, automotive components, etc., due to its flexibility, chemical resistance, and low cost [8].

2.2 STATISTICAL DATA ON PRODUCTION, CONSUMPTION AND MANAGEMENT OF POLYMER WASTE

Recent statistics indicate accelerated plastic production and the corresponding challenges in waste management. Global plastic production reached 400.3 Mt in 2022 and continued to grow to 413.8 Mt in 2023 [9].

Among types of plastic polymers, PE accounts for the largest share of total production – 26.2%, followed by PP at 19%. PVC (used mostly in construction) and PET (used for beverage packaging) account for 12.8% and 6.2% respectively; PS (used in protective packaging and insulation) 5%. The remaining 16% consists of other thermoplastics (such as ABS, polycarbonate, polyamides, etc.) and thermoset materials (epoxy resins) [9].

In Europe, nearly 40% of plastic is used for packaging, 20.4% in the construction sector (e.g., pipes, insulation, PVC windows, etc.), 8.8% in the automotive industry (for components, casings, interior finishes), 6.2% for electrical and electronic equipment, and 3.2% in agriculture (films, pots, irrigation systems) [10]. The remaining 21.4% is classified as “other” uses (including medical sector, furniture, various industrial products, etc.) [10].

2.3 CURRENT TECHNOLOGIES FOR VALORIZATION OF POLYMER WASTE

The recycling and recovery methods addressed in this work are classified into:

1. **Mechanical recycling**, which delays but does not completely avoid the final disposal of waste. Using recycled plastic reduces the amount of virgin plastic needed [11].
2. **Resource recovery or chemical recycling**, by breaking down the polymer structure of plastic to obtain the original monomers. These monomers can then be reused as raw material in producing new plastics [12]. The most studied and advanced technologies for resource recovery from plastic waste are: depolymerization, hydrocracking, gasification, and pyrolysis;
3. **Energy recovery**, i.e. incineration for energy valorization of plastic waste that cannot be recycled or whose recycling is not economically feasible.

2.4 CONVENTIONAL PYROLYSIS

Pyrolysis is the thermochemical decomposition of organic material at high temperatures in the absence of oxygen, in an inert atmosphere. Unlike combustion processes involving oxygen, pyrolysis relies on thermal decomposition, transforming waste into gaseous, solid, or liquid products. These can later be used for energy generation or as chemical feedstocks [13].

In general, the pyrolysis of materials is governed by the devolatilization process, which consists of the thermal breakdown of the material and the release of volatile compounds. The devolatilization of plastic is a complex phenomenon that can be summarized in three stages: initiation, propagation, and termination [14]:

1. **Initiation stage:** The supplied thermal energy causes random scission of C–C bonds in the polymer backbone (random chain scission) or, sometimes, end-chain scission, generating free radicals.
2. **Propagation stage:** The radicals thus formed initiate a chain reaction that leads to (i) hydrogen transfer (replacement of a hydrogen atom in a polymer molecule by a radical, generating a new radical species) and (ii) β -scission (secondary radicals break at the β position, forming olefins and regenerating primary radicals) [14], [15].
3. **Termination stage:** The existing radicals recombine to form stable molecules, thus terminating the reactive chains. These stable products no longer participate in the chain reaction and appear in the final composition of the obtained gases and oils [14], [16].

2.5 MICROWAVE-ASSISTED PYROLYSIS

Microwave-assisted pyrolysis is an advanced thermochemical conversion technology in which microwave (MW) energy is used to convert plastic waste into valuable products. The main difference between this technology and conventional pyrolysis lies in the heating mechanism, which enables volumetric heating of the material as opposed to predominantly surface heating (via conduction and convection) in conventional pyrolysis systems [17], [18]. This difference provides several advantages, such as a faster and more uniform temperature distribution, higher energy efficiency, and improved quality of final products [18].

Pure plastics generally have very low values of $\tan \delta$ (loss tangent). For example, PE, PP, and PET have $\tan \delta \sim 0.001\text{--}0.003$ [17], which are insufficient for efficient microwave absorption and conversion. Instead, materials like silicon carbide (SiC) and activated carbon have $\tan \delta$ on the order of $0.2\text{--}0.9$ [17], making them good microwave absorbers. To overcome this inherent limitation of plastic, dielectric materials—commonly called microwave susceptors—are used.

These absorbing materials serve to convert the microwave energy they absorb into thermal energy, which is then transmitted to the plastic via conduction, thus facilitating the pyrolysis reaction [1], [18]. The most common susceptors are activated carbon, graphite, biochar, SiC, and various metal oxides.

From a technical perspective, microwave-assisted pyrolysis exhibits a series of differences from conventional pyrolysis, both in terms of heat transfer mode and in terms of process performance and product quality. (For example, volumetric heating leads to reduced heat losses and potentially different product distribution compared to surface heating.)

3. PHYSICO-CHEMICAL CHARACTERIZATION OF LDPE WASTE

The material chosen for the experimental study consists of plastic waste generated in the industrial packaging sector, coming mainly from films used for wrapping pallets of various goods (such as electrical equipment, appliances, furniture, etc.). Thus, the waste investigated in this study is LDPE film.

In this chapter, several analytical methodologies are applied to the LDPE waste. Elemental (CHNS) analysis and proximate analysis are performed. Fourier-transform infrared spectroscopy (FTIR) is also conducted to identify characteristic functional groups and assess the degree of crystallinity. The calorific value of the waste is determined as an indicator of its energy potential, and the thermal behavior is investigated by thermogravimetric analysis (TGA), coupled with a kinetic study of the pyrolysis process. These complementary methods provide insight into the properties of the studied waste, forming the basis for optimizing the pyrolysis process.

3.1 GENERAL PROPERTIES OF LDPE

The density of LDPE at 20 °C ranges between 0.91 and 0.94 g/cm³ [19] a value determined by its molecular structure, which is characterized by a high degree of branching[52]. Mechanically, LDPE is distinguished by good impact resistance and high ductility, characteristics that allow the material to deform under mechanical stress without breaking [20], [21]. LDPE also exhibits good resistance to moisture and to many chemicals (acids, bases, alcohols, and esters). In addition, LDPE has low water vapor permeability, an important feature in protecting moisture-sensitive products [22], [23], [24]. In terms of thermal properties, LDPE has a melting point between 105 °C and 115 °C, and the temperature at which thermal degradation begins exceeds 440 °C.

3.2 CHARACTERIZATION OF LDPE WASTE

Elemental analysis indicated an average content of 85.3 wt% C and 13.4 wt% H, with traces of N (<0.2 wt%) and S (<0.001 wt%), values comparable to those of pure LDPE. Proximate analysis showed over 99.8 wt% volatile substances and only 0.18 wt% ash, suggesting the potential for almost complete conversion into condensable products/gaseous phase. FTIR spectroscopy confirmed characteristic bands at 718, 1458, 2854, and 2909 cm⁻¹, corresponding to the specific structure of low-density polyethylene, composed of methylene groups with methyl end groups. The degree of crystallinity was calculated to be 46.4% for the studied LDPE waste. The higher heating value measured with a bomb calorimeter characterizes this waste as having good energy potential, with values around ≈46 MJ/kg.

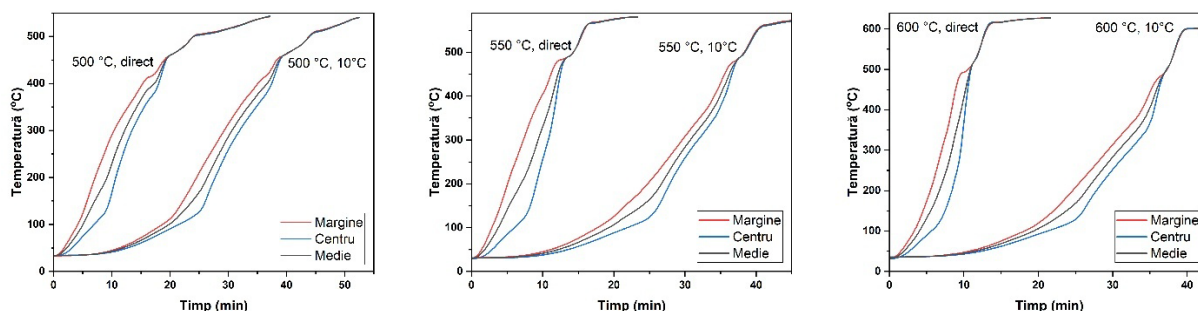


Figure 3.1. Temperature profile inside the LDPE sample at 500 °C, 550 °C and 600 °C for the two cases (direct exposure to the process temperature and exposure with a constant heating rate of 10 °C/min).

TGA studies conducted at 500 °C, 550 °C, and 600 °C revealed thermal degradation in a single main stage, with significant mass losses in the range of 450–500 °C. Regardless of the final temperature or heating mode (direct introduction to the target temperature vs. gradual heating at 10 °C/min), the LDPE decomposed almost completely, with total conversion to volatile products exceeding 99% of the initial mass. At the end of pyrolysis, the remaining solid residue was under 1 wt% (~0.3–0.5% at 500 °C and less than 0.1% at higher temperatures), confirming the proximate analysis results regarding the negligible content of non-volatile components[56].

The Coats–Redfern method showed that the F1 (first-order) and D3 (three-dimensional diffusion, Jander model) reaction models give activation energies comparable to literature values (~110 kJ/mol and ~170 kJ/mol, respectively) and high correlation coefficients ($R^2 > 0.997$) for all three isothermal regimes investigated. However, the high correlation coefficients ($R^2 > 0.99$) for several tested kinetic models indicate that the Coats–Redfern method alone cannot unambiguously identify the reaction mechanism, making additional methods necessary. The master plot analysis confirmed to some extent a diffusion mechanism for slow heating and a 2nd–3rd order reaction mechanism for rapid heating[57].

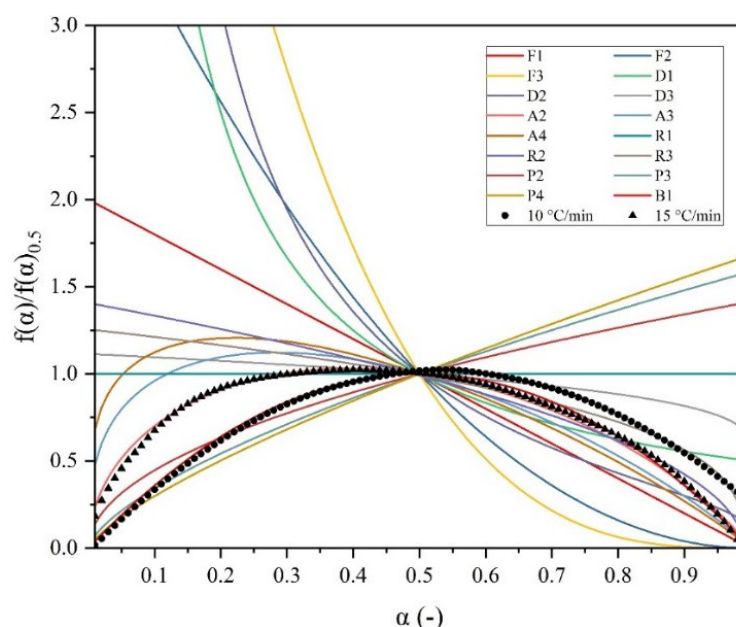


Figura 3.2. Fitting of the experimental curves for both heating rates with the theoretical “Master Plot” for 500 °C.

In conclusion, the investigated LDPE waste features: (i) suitable purity and volatility for advanced pyrolysis processes; (ii) a thermal degradation threshold between 450 °C and 500 °C; and (iii) a high calorific value for energy recovery. The data confirm the feasibility of thermally processing this waste stream and provide the foundation for optimizing pyrolysis processes in conventional or microwave-assisted reactors.

4. EXPERIMENTAL STUDIES ON THE CONVENTIONAL PYROLYSIS OF LDPE

This chapter examines the thermochemical behavior of LDPE under conventional pyrolysis, both in the absence and presence of catalysts. Additionally, the synergistic effect of co-pyrolysis of LDPE with other organic materials is investigated. The study of conventional pyrolysis was carried out using three experimental configurations:

- **Non-catalytic pyrolysis**, where two heating regimes (controlled ramp of 10 °C/min vs. direct exposure to the target temperature) were compared at three temperature levels (500, 550, and 600 °C) [59].
- **Catalytic pyrolysis at 600 °C with a heating rate of 10 °C/min.** LDPE was pyrolyzed in the presence of two catalysts (BSTZc and HZSM-5) placed on a quartz fiber support [59].
- **LDPE/biomass co-pyrolysis.** The biomass selected for the co-pyrolysis study was agro-industrial chicken bone waste [60].

4.1 NON-CATALITICĂ PROCESSING LDPE VIA NON-CATALYTIC PYROLYSIS

In this section, pyrolysis was studied at 500 °C, 550 °C, and 600 °C under two heating regimes: controlled heating at 10 °C/min and direct exposure to the process temperature. Configurația experimentală este reprezentată schematic în Figura 4.1. The experimental setup is schematically represented in Figure 4.

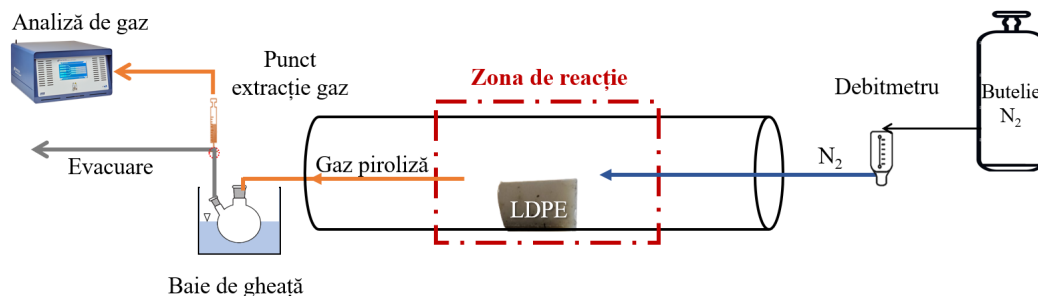


Figura 4.1. The experimental setup is schematically represented

The distribution of products from LDPE pyrolysis shows a significant influence of both the process temperature and the heating mode. In the case of a constant heating rate of 10 °C/min, a gradual reduction of the solid fraction (wax) is observed from 66.3 wt% at 500 °C to 44.8 wt% at 600 °C, accompanied by an increase in the gas fraction from 33.7 wt% to 55.2 wt%. A similar trend is seen for direct exposure to the final temperature, where the wax decreases from 60.1 wt% (500 °C) to only 38.0 wt% (600 °C).

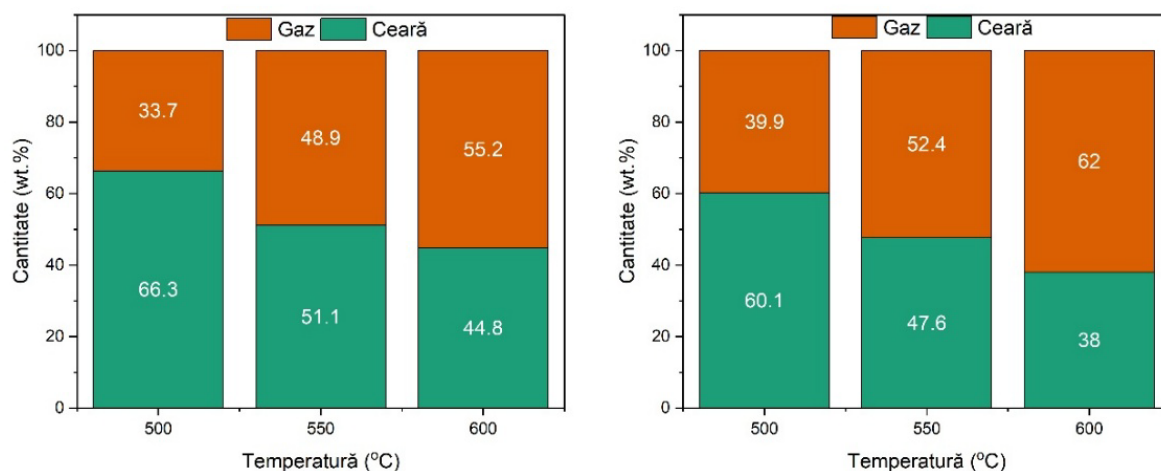


Figura 4.2. Product distribution for the two types of experiments (gradual heating – left and direct exposure – right).

4.2 PROCESSING LDPE VIA CATALYTIC PYROLYSIS

The use of BSTZc and HZSM-5 catalysts in LDPE pyrolysis significantly influences the distribution of reaction products. In the absence of catalysts, a typical distribution for LDPE is observed, consisting of wax (44.8%) and gas (55.2%). Introducing the BSTZc catalyst into the pyrolysis process results in a significant increase of the wax fraction to 60.1% and a decrease of the pyrolysis gas to 39.9%. This suggests a moderate catalytic activity that favors the production of heavier hydrocarbons by limiting complete cracking of polymer chains. The use of HZSM-5 led to the highest wax proportion, 81.3%.

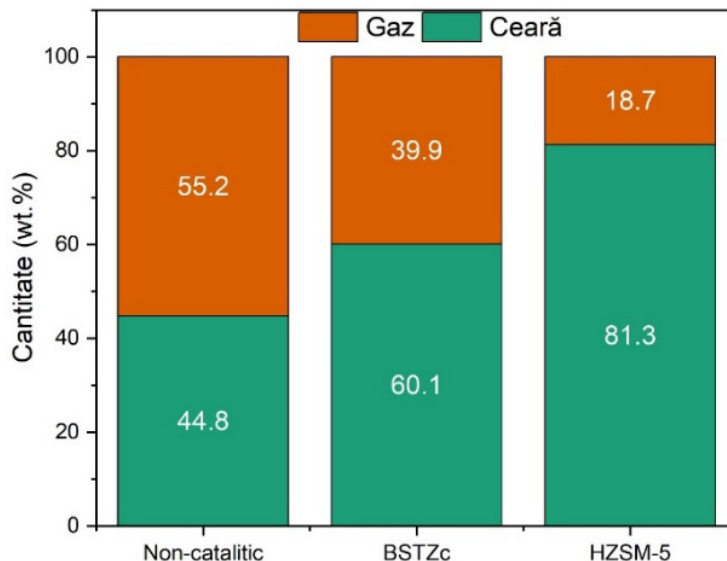


Figura 4.3. Product distribution in the case of catalytic pyrolysis.

4.3 PROCESSING LDPE VIA CO-PYROLYSIS WITH BONE WASTE

In the co-pyrolysis study, LDPE waste was used in flake form, and chicken bone waste was used in 3 forms: dried chicken bone waste (L3), biochar from bone waste (C3), and ash from bone waste (A3).

The process gas composition is significantly influenced by the type of material used in co-pyrolysis, according to Table 4.1. In all three co-pyrolysis experiments, CO₂ and CO are the

predominant components in the gas (28–44 vol% CO₂ and 9–20 vol% CO). As the bone waste is thermally pre-treated (L3 → C3 → A3), the CO₂ content drops by approximately 15 percentage points, while the CO content increases (from 8.5 vol% up to a maximum of 20.45 vol% for C3). A similar increasing trend is observed for O₂, which can be attributed to the participation of inorganic fractions (mainly Ca-based compounds) in deoxygenation reactions, resulting in a higher-quality liquid/wax fraction.

Tabel 4.1. Average composition of co-pyrolysis gases from LDPE and L3/C3/A3

| Component (vol.%) | L3 | C3 | A3 |
|--------------------------|-----------|-----------|-----------|
| H ₂ | 4,00 | 10,66 | 1,71 |
| O ₂ | 1,27 | 3,32 | 4,88 |
| CO | 8,51 | 20,45 | 16,88 |
| CH ₄ | 8,19 | 5,45 | 5,22 |
| CO ₂ | 43,72 | 28,62 | 33,31 |
| Ethylene | 6,03 | 9,03 | 12,43 |
| Ethane | 10,71 | 10,81 | 11,96 |
| Propene | 4,42 | 3,07 | 3,48 |
| Propane | 5,23 | 5,13 | 6,85 |
| Butane | 6,15 | 1,32 | 0,64 |
| Pentane | 0,80 | 0,95 | 1,13 |
| Hexane | 0,96 | 1,18 | 1,51 |
| Total hydrocarbons | 34,31 | 31,49 | 38,00 |

5. LDPE EXPERIMENTAL STUDIES ON MICROWAVE-ASSISTED PYROLYSIS OF LDPE

Chapter 5 focuses on the experimental investigation of LDPE microwave-assisted pyrolysis, pursuing objectives related to process efficiency and optimization of reaction products. The aim of this chapter is to identify and validate the optimal conditions under which microwave energy can be used to convert LDPE waste into valuable products, maximizing conversion and controlling the distribution of resulting fractions (gas, liquid, solid).

A first objective was to evaluate the thermal behavior and yields of LDPE pyrolysis in a microwave field at 2.45 GHz, 200 W, in different configurations: without catalyst and in the presence of two distinct catalysts (one biochar-based catalyst doped with silicon, titanium, and zinc oxides, and a reference HZSM-5 zeolite catalyst).

The second important objective was scaling up the study to a higher experimental scale by performing microwave-assisted pyrolysis experiments at 400 W and 915 MHz on larger quantities of LDPE, in order to verify the reproducibility of results, process stability, and the impact of intensified conditions on product distribution. To achieve this, efforts were made to improve process efficiency by optimizing the susceptor material (through physical and chemical activation of biochar) and by developing new bio-catalysts for in-situ and ex-situ process stages.

Methodologically, a microwave pyrolysis system with controllable power and frequency was used to achieve these objectives. Additionally, susceptor materials and catalysts that were synthesized and characterized beforehand were employed. The reaction products were analyzed quantitatively and qualitatively, by evaluating the mass balance and the composition of the pyrolysis gases.

5.1 STUDY ON THE SELECTION OF SUSCEPTOR MATERIAL

The biochar was obtained by pyrolysis of bone waste at temperatures of 500 °C, 600 °C, and 700 °C with two heating rates (10 °C/min and direct exposure to the process temperature [25], [26]).

For testing microwave absorption, a Sairem MiniFlow 200SS reactor designed for laboratory activities was used. The reactor is equipped with a continuous microwave generator operating in TE₁₀ mode (transverse electric). The solid-state microwave generator operates in the 2.43–2.47 GHz frequency range and allows power adjustments from 0 to 200 W, in 1 W increments.

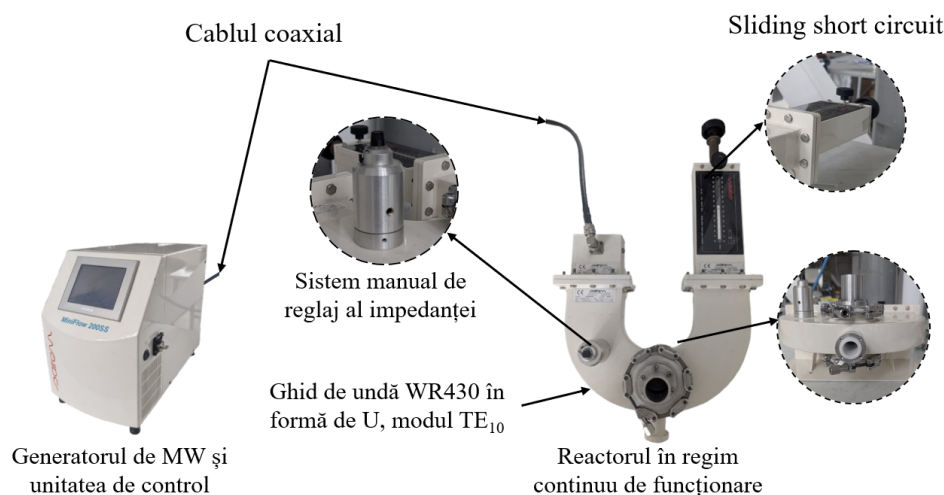


Figura 5.1. Sairem MiniFlow 200SS microwave installation and its main components.

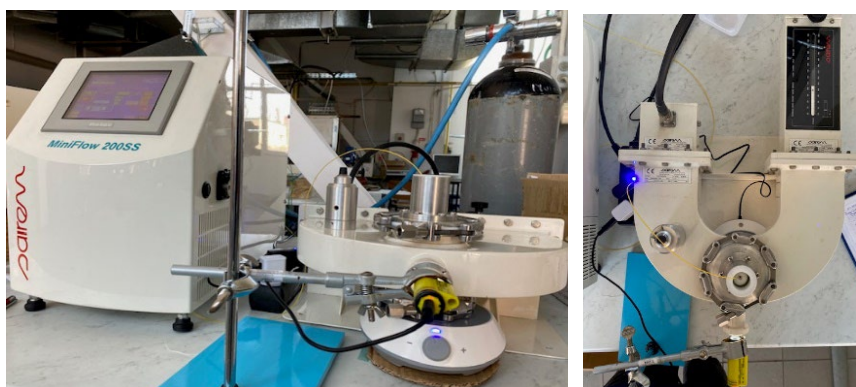


Figura 5.2. Experimental setup used for testing the biochar.

Following the experimental studies presented in Chapter 5, a series of results were obtained regarding the conversion of LDPE waste by microwave-assisted pyrolysis. It was demonstrated that this technology allows almost complete conversion ($\sim 100\%$) of LDPE into useful products, with the distribution of resulting fractions depending on the presence and nature of catalysts used. In the absence of catalysts, the process generates a significant amount of solid product in the form of wax (uncracked residue), indicating incomplete degradation of the polymer. By contrast, using a Ni/Fe-doped activated biochar catalyst (in-situ configuration) led to a substantial increase in the gas fraction (up to $\sim 72\%$ of reaction products, compared to $\sim 40\%$ under non-catalytic conditions) and an almost complete reduction of wax ($< 5\%$). This catalyst also promoted the formation of a pyrolysis gas rich in hydrogen (H_2 content $\sim 30\text{--}43\text{ vol}\%$ in the non-condensable gas), highlighting the potential of utilizing plastic waste for hydrogen production. On the other hand, acidic zeolite catalysts (HZSM-5) or ash-based Ni-impregnated catalysts (ex-situ configuration) favored the production of higher condensable fractions. For example, in the presence of the HZSM-5 catalyst, $\sim 74\%$ liquid + wax fraction was obtained (compared to 39% without catalyst), while using Ni-impregnated bone ash led to a distribution of approximately 56% gases, 33% oil, and 11% wax. The sequential combination of both types of catalysts (biochar/Ni/Fe in-situ + ash/Ni ex-situ) had a synergistic effect, almost completely eliminating the wax ($\sim 1\%$) and simultaneously yielding substantial gas ($\sim 58\%$) and liquid ($\sim 41\%$) fractions.

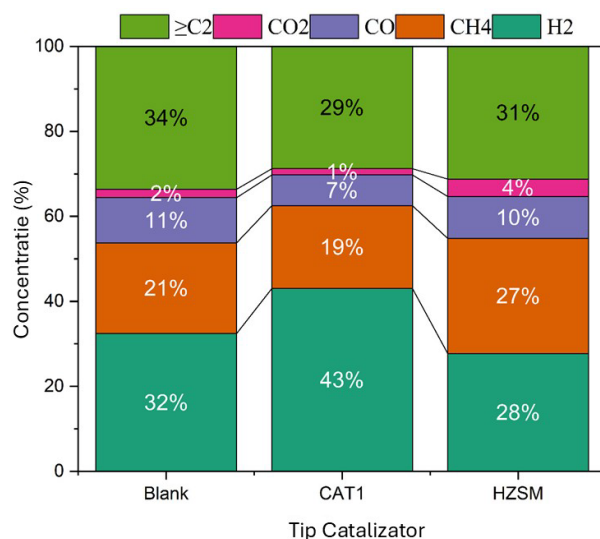


Figura 5.3. Composition of the MW-assisted pyrolysis gas of LDPE.

These results demonstrate that, through appropriate choice of catalysts and process configuration, LDPE microwave-assisted pyrolysis can be steered either towards obtaining a fuel (syngas) rich in energetic compounds (H₂, CH₄, C₂+ hydrocarbons, etc.) or towards obtaining oil, depending on the desired applications. Furthermore, increasing the microwave power from 200 W to 400 W (and shifting to 915 MHz frequency) proved beneficial: it enabled processing a quantity of LDPE 20 times larger in a shorter time (30–60 minutes) without loss of yield. Optimizing the experimental setup (including replacing the biochar susceptor with a more efficient graphite susceptor) helped to reach higher process temperatures (~600 °C) and maintain a stable reaction regime, confirming the feasibility of scaling up the process to a larger laboratory scale. In addition, chemical activation of the biochar used as susceptor showed that its properties can be significantly improved (increased surface area and porosity), which was reflected in better catalytic performance, while physical activation (with CO₂) highlighted the limitations of a material with low carbon content (leading to excessive consumption of the carbon phase and predominantly producing ash).

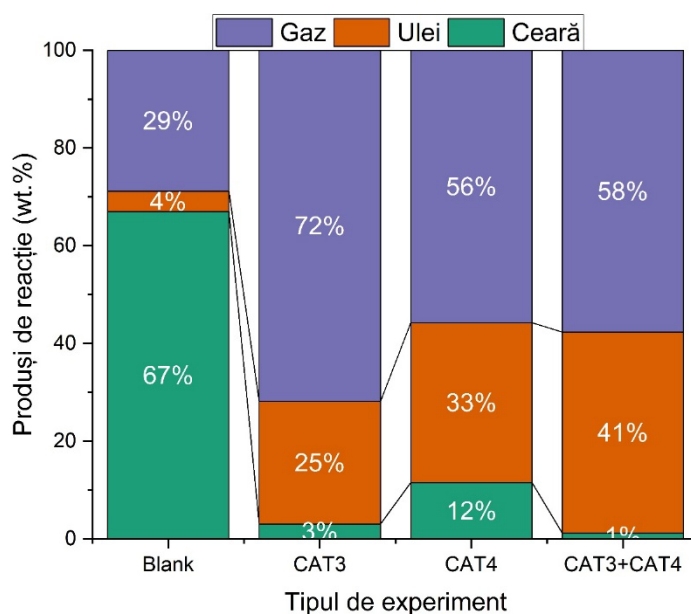


Figura 5.4. Distribution of reaction products for the 4 types of experiments, MAP 400 W.

6. BILANȚUL ENERGETIC AL PROCESELOR DE CONVERSIE UTILIZATE

Chapter 6 aims to perform an energy analysis of the pyrolysis process applied to LDPE waste, both in conventional and microwave-assisted regimes. The purpose of evaluating the energy balance is to determine the extent to which the chemical energy contained in the polymer waste can be recovered in the form of products with energy value, and the amount of energy required to carry out the pyrolysis process. Thus, the goal is to determine the overall conversion efficiency of the two pyrolysis regimes.

The first objective is to determine the calorific values of the main pyrolysis products (gas, oil, and wax) as an indicator of the energy potential of each fraction. This involves directly measuring, via calorimetry, the calorific value of the obtained oil and wax, as well as calculating the calorific value of the pyrolysis gas based on its composition. Using these data, the total energy recovered in the reaction products is calculated by summing the energy contributions of all fractions. By comparing the recovered energy with the initial energy content of the waste, one can estimate the energy yield of the conversion (the percentage of the waste's energy converted into useful products) and identify energy losses (for example, energy remaining unrecovered in solid residues or lost as heat).

The second objective is to estimate the overall energy efficiency of the process, defined by the ratio of energy obtained in products to the energy consumed to power the process. In this respect, the energy consumptions of the process are determined, including the heat required to heat and decompose the LDPE, the energy absorbed by the materials used, and heat losses to the system and environment. The energy consumption of the pyrolysis systems was estimated including both the process component (energy needed to heat and react the LDPE to complete conversion) and the technical component (energy consumed to heat the equipment and losses to the environment). For the conventional pyrolysis, the thermal input of the furnace was considered, and for the microwave-assisted pyrolysis, the power absorbed by the material was evaluated.

The energy analysis highlights the high potential for energy recovery from LDPE waste through pyrolysis, as well as the performance differences between the conventional and microwave-assisted regimes. The calorific values of the obtained products proved to be high, confirming that the pyrolysis fractions can be used as fuels. The condensable fractions (wax and oil) have higher heating values around 44–46 MJ/kg, close to the energy value of the original polymer and in line with literature data. The pyrolysis gas also has a significant energy content. It was found that the heating regime and the presence of catalysts influence the gas's energy quality: for example, in microwave-assisted pyrolysis with an acidic catalyst (HZSM-5), the resulting gas is greatly enriched in combustible hydrocarbons and has a volumetric calorific value of ~70 MJ/m³, almost double that of the gas obtained without catalysts or in conventional co-pyrolysis, where the presence of CO₂ energetically dilutes the mixture[86][87]. Overall, the products obtained in both regimes (gas, oil, wax) possess high calorific values comparable to conventional fuels, confirming that the chemical energy of the polymer waste can be efficiently converted into energy products.

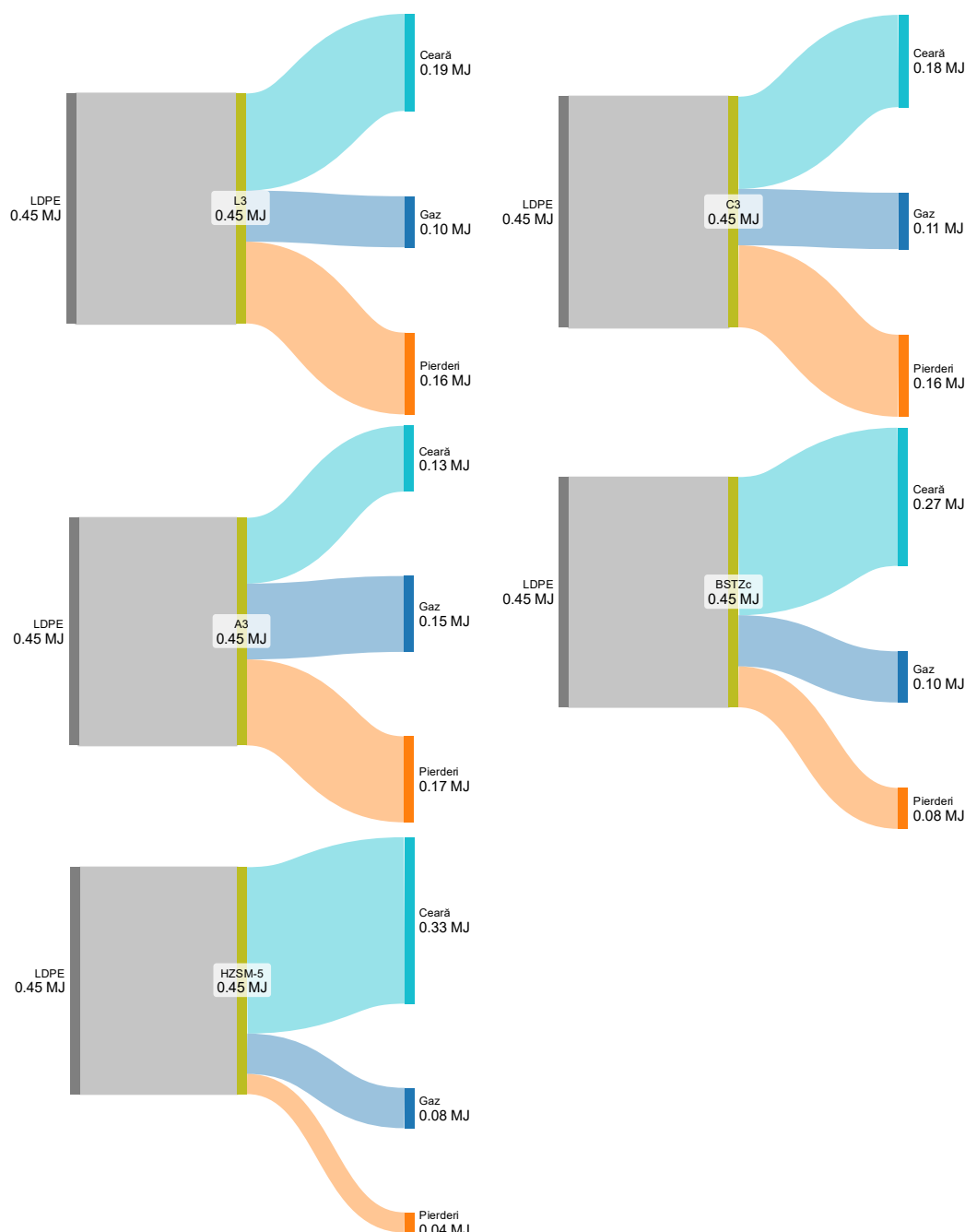


Figura 6.1. Sankey diagram of LDPE energy conversion – reactant and reaction products for conventional pyrolysis experiments

From the perspective of overall energy conversion efficiency, both in the conventional system and the microwave system, a large proportion of the initial LDPE energy was recovered in the reaction products. Under optimal conditions, the energy yield exceeded 90%. For example, in microwave-assisted pyrolysis experiments, all tested configurations recovered at least ~92% of the plastic's initial energy, reaching up to ~99% in the presence of the optimal combination of catalysts. This indicates minimal energy losses through residues or products with no energy value. In the absence of catalysts, or in co-pyrolysis with bone waste biomass, the conversion efficiency decreased, suggesting that part of the potential energy was lost either in forming non-combustible residues with negligible energy content or as unrecovered heat. Nevertheless, even in these cases, the energy balance remained favorable, demonstrating the process's capability to recover energy from LDPE.

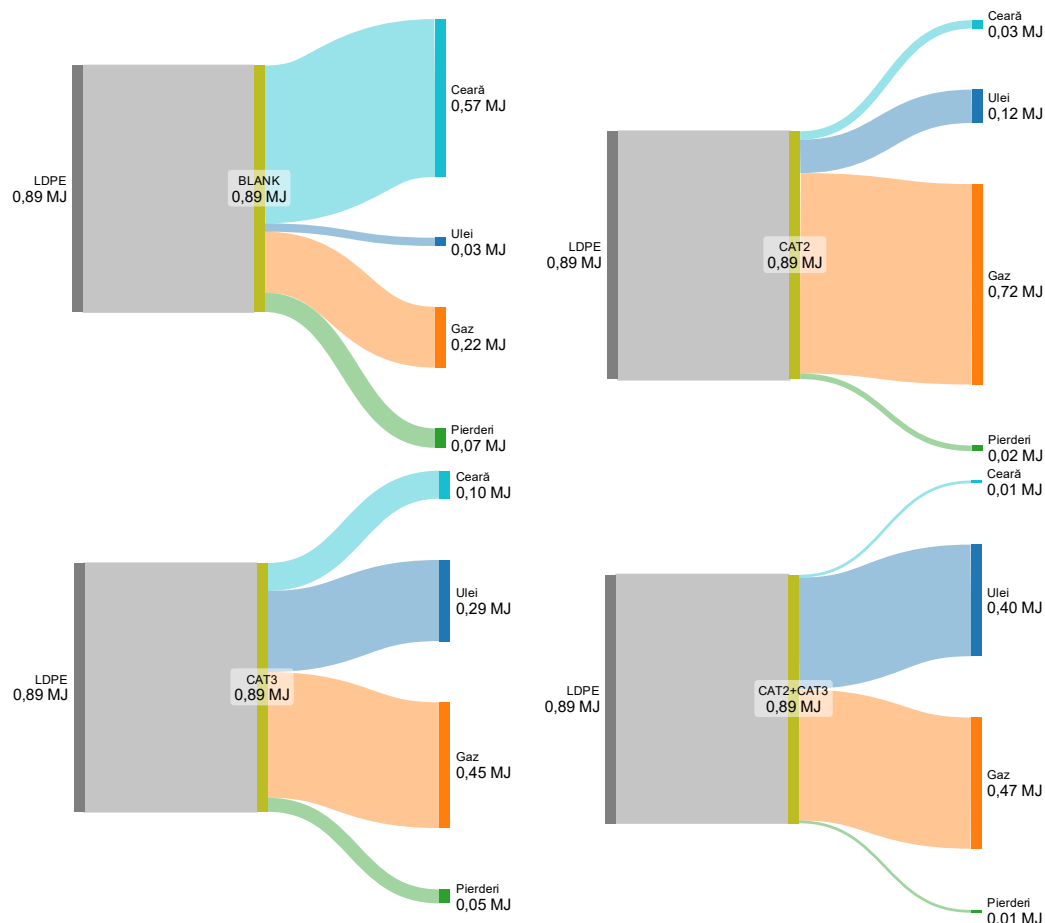


Figura 6.2. . Sankey diagram of LDPE energy conversion into reaction products for MAP 400 W experiments

It was observed that microwave-assisted pyrolysis offers advantages over the conventional process in terms of the efficiency of process energy use. Volumetric heating by microwaves, as opposed to classic external thermal heating, reduces losses and ensures a more efficient conversion of electrical energy into useful heat within the susceptor material. Therefore, the ratio of energy recovered to energy consumed is higher in the microwave-assisted regime (especially with catalysts), translating into a net better energy yield of the process.

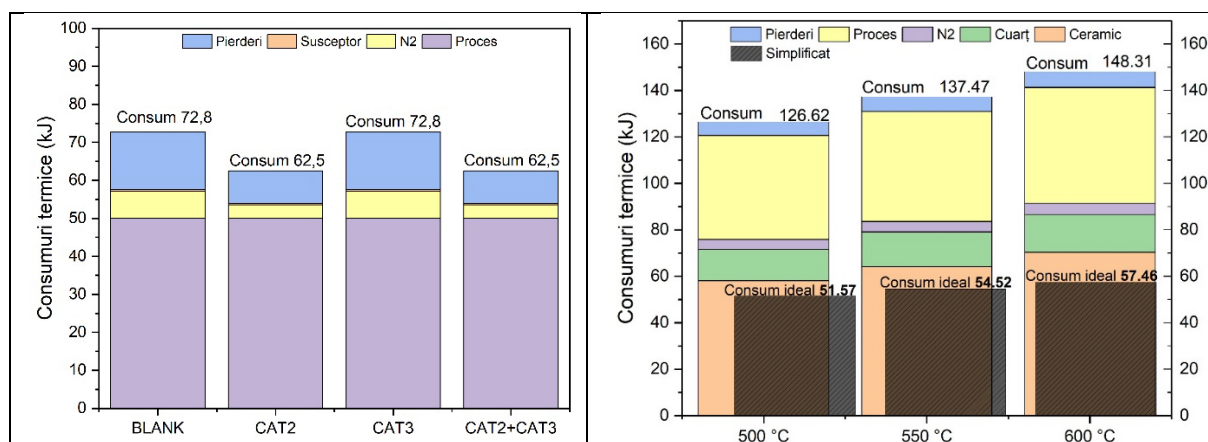


Figura 6.3. Specific thermal energy consumptions for the experimental pyrolysis setup in conventional and microwave-assisted systems.

7. GENERAL CONCLUSIONS

7.1 OBTAINED RESULTS

This work demonstrated the feasibility and efficiency of converting LDPE via pyrolysis, highlighting the advantages of both the conventional process and the microwave-assisted process. The detailed physico-chemical characterization of the LDPE waste (industrial packaging film) indicated an almost exclusively carbon (~85.3 wt% C) and hydrogen (~13.4 wt% H) composition, with negligible presence of other elements (traces of N and S <0.2 wt%)[92]. The waste has a very low ash content (~0.18 wt%) and over 99.8 wt% volatile matter, which suggests the potential for almost complete conversion of the polymer mass into reaction products (oils and gases), with negligible solid residue. Moreover, because of the absence of heteroatoms (chlorine, oxygen, nitrogen) in the LDPE structure, no toxic or corrosive secondary compounds are generated; the resulting pyrolysis oil has a predominantly aliphatic composition (paraffins, olefins) and a high calorific value of ~39–40 MJ/kg, comparable to fossil fuels. The investigated waste has a crystallinity degree of ~46% and a higher heating value of ~46 MJ/kg, similar to the pure polymer.

From a process performance perspective, the experiments showed that thermal pyrolysis of LDPE leads to almost complete devolatilization (>99%) of the material, with the peak of thermal degradation in the range of 450–500 °C. Both the conventional regime (convective heating in a furnace) and the microwave-assisted regime (internal volumetric heating) achieved conversion of the majority of the waste mass into useful products. In the absence of any catalyst, the formation of a significant solid fraction in the form of wax (uncracked heavy paraffins) was observed, indicating incomplete cracking of LDPE polymer chains.

Comparing the conventional regime to the microwave-assisted regime revealed differences in thermal efficiency and conversion. Microwave heating (2.45 GHz and 915 MHz, respectively) provides volumetric heating, avoiding the extra heat inputs and losses to the surroundings that are characteristic of classical heating. Thus, a superior energy yield was found in the microwave-assisted system – the ratio of energy recovered in products vs. energy consumed was more favorable than in conventional pyrolysis. Additionally, increasing the microwave power (from 200 W to 400 W) and using the 915 MHz frequency allowed processing ~20 times more LDPE in a 30–60 minute time span without a decrease in conversion yields. Optimizing the experimental configuration at 915 MHz by replacing biochar with a more thermally efficient graphite susceptor led to achieving temperatures of ~600 °C inside the sample mass and maintaining a stable pyrolysis regime, demonstrating the feasibility of scaling up the process to a larger laboratory scale. Calorimetric and chromatographic analyses of the obtained products confirm that all pyrolysis fractions have high energy potential: the oils and waxes obtained have higher heating values of ~44–46 MJ/kg, values comparable to the energy content of the initial polymer, and the pyrolysis gases have significant energy content, especially when their composition is improved through the use of catalysts. The chemical energy contained in the LDPE waste was efficiently converted into valuable energetic products (gas, oil, wax), and the energy recovery efficiency into products exceeded 90% in both operating regimes. Under optimized conditions, a recovery of ~99% of the initial LDPE energy in the form of combustible products (pyrolysis oil and gas) was even achieved when the most effective catalytic combination was used. Even without catalysts or in conventional co-pyrolysis with bone waste, the energy conversion efficiency remained above ~60–65%, with energy losses attributable to the formation of non-combustible residues (e.g., ash) and thermal losses. These findings show that LDPE pyrolysis (both classical and microwave-assisted) is a viable energy recovery

method, capable of converting plastic waste into liquid and gaseous fuels with high efficiency and recovering the majority of the energy contained in the waste.

7.2 ORIGINAL CONTRIBUTIONS

Although this thesis studied a topic well documented in the specialized literature, its original contributions lend a degree of novelty and scientific interest to the work.

1. While methods for characterizing LDPE waste are standard in this field, the original contribution lay in scaling up the thermogravimetric analysis and kinetic process study to a macro level. In the literature, most such investigations are conducted using specific analytical instruments (TG analyzers) that limit the sample size to the microgram scale. In this study, the order of magnitude for these analyses was ~20 grams. This larger scale provides complex information on the kinetic degradation of the waste, which would have been more difficult to identify otherwise. Furthermore, in the macro-thermogravimetric analysis of this study, by using a thermocouple embedded in a solid LDPE cylinder, it was possible to measure the internal temperature of the sample in real-time, rather than the reaction zone temperature. Thus, it was possible to identify characteristics related to heat transfer inside the LDPE. This approach is geared towards studying the thermal behavior of LDPE at an industrial scale, where large continuous quantities of sample behave as a whole, and not as fine particles in the microgram range.
2. By introducing optimization studies for the conventional pyrolysis process of LDPE, the synergistic transformation mechanism in the presence of biomass materials derived from chicken bone waste was investigated. Co-pyrolysis of lignocellulosic biomass/plastic is well documented in the literature, but this study proposes a new material with potential to improve process performance by using this type of waste. The co-pyrolysis study was carried out with different preparation stages of the bone waste (as-is, converted to biochar, and to ash) to identify synergistic interactions between these two materials. In addition, by using this type of waste as a co-pyrolysis material, a new method of managing agro-industrial waste is suggested.
3. In this work, new materials were used, synthesized specifically for the purpose of recovering LDPE waste via pyrolysis, such as: susceptor materials made from biochar obtained from chicken bone waste, and bio-catalysts based on the same type of bone waste. These were synthesized using innovative microwave-ultrasound assisted methodologies. Moreover, the catalyst synthesis was designed with a dual role in mind: to function as both microwave susceptor and cracking catalyst for LDPE. Thus, these catalysts were studied, tested, and characterized, and their dual influence on the pyrolysis process was evaluated[102][103].
4. The efficiency improvements of the pyrolysis processes were also based on proprietary processing configurations, by using bio-catalysts in in-situ, ex-situ, and combined systems. Additionally, in the 400 W microwave-assisted pyrolysis, a new experimental setup was employed, consisting of designing and constructing a quartz reactor and positioning each element within the MW cavity (LDPE waste, susceptor material, quartz reactor, catalyst, condensation system) in an optimized way.
5. The analysis of energy consumption was based on proposed assumptions and calculations to highlight the actual energy consumption of the pyrolysis process, depending on the type of process configuration and the equipment used.

7.3 DEVELOPMENT PERSPECTIVES

1. Expanding the study to mixtures of polymer waste.

A potential direction for extending the thesis is the application of the developed research methodology to mixtures of polymer waste, in order to quantify the thermo-physico-chemical transformations in pyrolysis processes. Additionally, typical contaminants present in such waste (e.g., organic residues, moisture, inorganic additives) could be included as variables in the study to determine their impact on transformation mechanisms, reaction products, and the energy efficiency of the processes. Extending the study to real waste, such as plastic fractions from municipal garbage or agricultural waste (contaminated greenhouse films, mixed packaging), is feasible and may increase the industrial applicability of the conversion solutions

2. Numerical modeling and process simulation.

Complementary to the experimental study, developing a numerical model of the LDPE pyrolysis process would allow a deeper understanding of the phenomena and help optimize the process. New detailed kinetic models could be developed, calibrated on the basis of existing experimental data, to estimate the distribution of products as a function of process conditions. Likewise, a comprehensive analysis of heat and mass transfer within the LDPE sample could be performed. In addition, computational fluid dynamics (CFD) simulations of the microwave reactor could provide information on the distribution of the electromagnetic field and temperature in the material, aiding in the design of a larger-scale reactor (optimal cavity configuration, susceptor position, geometry, and size, etc.). At the same time, integrating data into an energy and economic balance model would allow assessment of industrial viability (energy efficiency, operating costs, carbon footprint of the process compared to other waste management methods).

7.4 SCIENTIFIC CONTRIBUTIONS

7.4.1 Published scientific articles

1. **Macavei, M. G.**, Gheorghe, C., Ionescu, G., Volceanov, A., Pătrașcu, R., Mărculescu, C., & Magdziarz, A. (2024). Thermochemical Conversion of Animal-Derived Waste: A Mini-Review with a Focus on Chicken Bone Waste. *Processes* (Vol. 12, Issue 2). <https://doi.org/10.3390/pr12020358>
2. Ionescu, G., **Macavei, M.**, Pătrașcu, M., Volceanov, A., Pătrașcu, R., Werle, S., Mlonka-Mędrala, A., Elena Coman, A., Magdziarz, A., & Mărculescu, C. (2025). New integrated processing of chicken bone waste using an enzymatic pretreatment and slow pyrolysis to produce green chemicals. *Energy Conversion and Management*, 323, 119281. <https://doi.org/10.1016/J.ENCONMAN.2024.119281>
3. Sandu, M. R., Boldor, D., **Macavei, M. G.**, Magdziarz, A., & Mărculescu, C. (2025). Heat and flow dynamics in biomass reactors under pyrolysis conditions: Computational insights. *Renewable Energy*, 244. <https://doi.org/10.1016/j.renene.2025.122691>

7.4.2 Scientific articles accepted for publication

1. **Macavei, M. G.**, Pătrașcu, M., Ionescu, G., Gheorghe, C., Petcu, G., Volceanov, A., Magdziarz, A. Mărculescu, C. (2025). Bio-based material as microwave susceptor and catalyst support for plastic waste microwave-assisted pyrolysis. *Clean Technologies and Environmental Policy*

2. **Macavei, M. G.**, Ionescu, G., Mărculescu, C. (2025). Kinetic investigation of low-density polyethylene pyrolysis: effects of heating rate. SCIENTIFIC BULLETIN, Seria C: Inginerie Electrică și Știința Calculatoarelor
1. **Training internships**
2. **Mobilitate ERASMUS+** for 4 months, Silesian University of Technology, Gliwice, Polonia
3. **Winter School** „Smart Public Space in Prosument Energy Transition”, Silesian University of Technology, Gliwice, Polonia

7.4.3 Member in research projects

1. „Green chemistry and thermochemical processing, a convergent approach towards biobased chemicals and hydrogen synthesis –**CONVERGREEN**” NO. CF 86/15.11.2022, COD 86 (06/2023-prezent).
2. Tehnologii curate de procesare și/sau valorificare materiale cu potențial combustibil – **CLEANTECH**”, ID 105958. (08/2023 – 10/2023)

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