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DEPARTMENT OF BIORESOURCES AND POLYMER SCIENCE

DOCTORAL THESIS

PYROLYSIS OF BIOMASS OR PLASTIC WASTE

Piroliza Deşeurilor de Biomasa sau de Plastic

Thesis summary

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CHAPTER II

RESEARCH OBJECTIVES

The general objective of this doctoral thesis is to investigate the catalytic pyrolysis process of polyolefins (polypropylene and low-density polyethylene), coming from both commercial sources (granules), waste plastics (end-of-life automotive components, waste polyethylene films), as well as the representative lignocellulosic residue – lignosulfonate, derived from the cellulose industry. The research aims to obtain valuable energy and chemical products in order to reduce the volume of non-recyclable waste and to promote sustainable and circular solutions for the recovery of materials and energy. In this context, the thesis combines a complex experimental approach with a comparative analysis between pyrolysis technologies based on conventional heating and microwave-assisted heating, following the influence of catalysts and working conditions on the yield and composition of the resulting products.

A complementary direction of the research is aimed at the application of catalytic pyrolysis on biomass, by studying the behavior of lignosulfonate as a renewable raw material. This extension brings added relevance to the work, allowing the comparison of the performances and mechanisms of thermal degradation between fossil and biogenic sources.

In order to achieve the general objective, the following research directions have been defined:

- ✓ The analysis of the pyrolysis products will be carried out by separating the liquid fractions in a pilot plant equipped with a distillation column with adiabatic operation, obtaining light (gasoline type), medium (diesel type) and heavy fractions. Their composition, together with that of the gases, was determined using techniques such as GC-FID and GC-MS, and for the gas phase the gross caloric values were calculated in order to evaluate the energy potential.
- ✓ The plastic waste resulting from car dismantling will be characterized by physico-chemical methods;
- ✓ Comparison between the performance of pyrolysis plants with conventional heating (electrical resistance) and microwave-assisted ones, using an adjustable single-mode cavity. Parameters such as energy efficiency, reaction time and process control will be tracked, highlighting the advantages and limitations of each heating method.
- ✓ For the pyrolysis of plastics, affordable catalysts such as modified clinoptilolite (e.g. AMD1, AMD4), bentonite, activated carbon, and for lignocellulose pyrolysis Mo/Al_2O_3 and $NiMo/Al_2O_3$ catalysts will be used. The effect of catalysts on the composition of the resulting gases and liquids will be evaluated. In particular, the influence on the selectivity towards light hydrocarbons (C_5–C_{13}) in the liquid fraction and on the H_2 and CH_4 content in the gas phase will be followed.
- ✓ The yields for the gaseous, liquid and solid phases obtained from pyrolysis will be determined. Based on the composition and calorific value of the gas mixtures produced, the potential for their direct use in motor generators for the cogeneration of electricity and heat was estimated.
- ✓ The research will use plastic waste from the automotive industry, waste such as foils. The behavior of these materials in conditions similar to industrial ones was followed, using a reactor with a capacity of 3 kg, as well as in a reactor with the application of microwaves, evaluating the feasibility of integration into a complex recovery flow.

- ✓ The thermal behavior of lignosulfonate, a lignocellulosic residue, will be investigated under thermocatalytic pyrolysis conditions, using synthetic catalysts. The composition of the products obtained will be analyzed.

CHAPTER III

PYROLYSIS OF PLASTIC WASTE IN LABORATORY PROTOTYPE PLANT

3.1. Materials and methods

In these experiments, clean polyethylene granules (LDPE MI 2 from Romcolor, Copăceni, Romania) and polyethylene film waste from the company IRIDEX (Bucharest, Romania) were used.

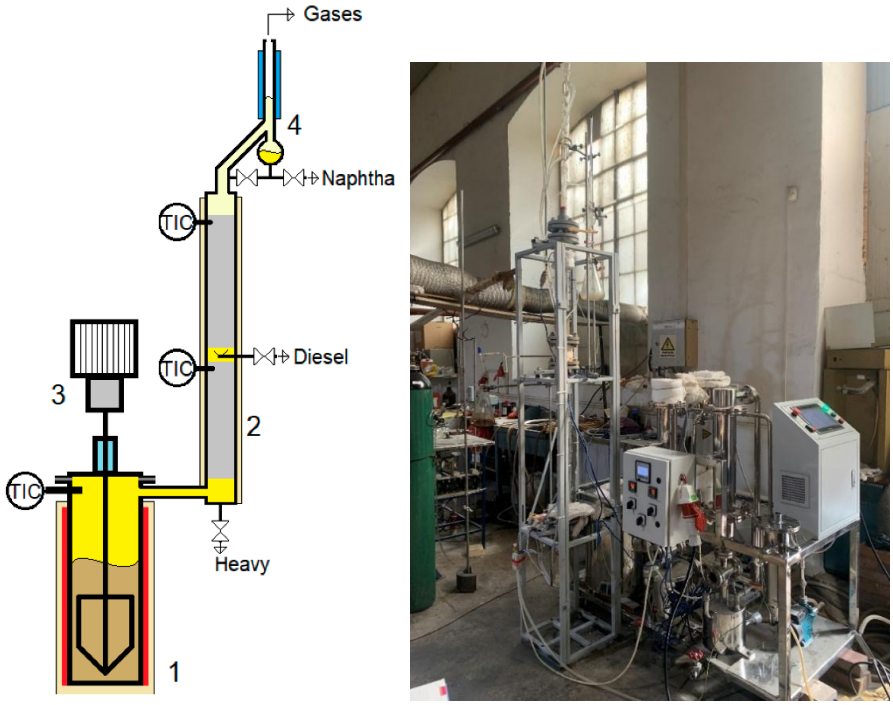
Three types of catalysts were used in the pyrolysis process, with particle sizes smaller than 200 μm: bentonite catalyst (Bentonite), with the chemical formula $Al_2O_{34}SiO_2H_2O$, and two clinoptilolite zeolite catalysts (AMD1 and AMD4) with the chemical formula $(Na,K,Ca)_{2\text{-}3}Al_3(Al,Si)_2\ Si_{13}O_{36}\text{-}12H_2O$, from AMD INITIATIVE SRL, Bucharest, Romania.

The zeolytic catalysts used, AMD1 and AMD4, were modified using the following procedures: 1. Physical treatments; 2. Heat treatments; 3. Chemical treatments; 4. Treatments for selection and enrichment in the crystalline phase.

The polypropylene (PP) raw material used in this study was obtained from car dismantling operations, in particular from car interior components such as the car pillar. The material was provided by SC Pieseauto Dez SRL, Constanța, Romania. No cleaning or washing has been performed; The plastic was only cut by hand into small pieces to facilitate uniform heating and contact with the catalyst during pyrolysis.

Two catalytic materials were used in this study: activated carbon (AC) and a synthetic zeolite-type catalyst, designated AMD4. The activated carbon, derived from coconut shell, 6*12 mesh, purchased from Legend Inc. USA, was used at concentrations of 5% by weight, 10% by weight and 20% by weight relative to the polypropylene feedstock, with additional tests involving the reuse of activated carbon recovered from previous cycles. AMD4 zeolite, characterized by an area of 147.00 m^2/g, an acidity of 0.6300 mEq/g and a porosity of 0.119 cm^3/g, was applied at a concentration of 20, 10 and 5 % by weight. An experimental setup also combined 5 wt% AC with AMD4 5% wt.

The equipment used was a prototype laboratory installation coupled with a distillation column. The experimental setup is described in Figure 0-1.



A B

Figure 0-1. (A) Experimental plant for plastic pyrolysis: 1-pyrolysis reactor with feeding flange and temperature control; 2-column two-section distillation; 3-stirring system (variable speed electric motor, speed reducer, sealing box, stirrer); 4-upper capacitor with reflux divider; (B) image of the experimental pilot installation



A B C

Figure 0-2. (A) Head distillation column; (B) Distillation column, heated with three elements; (C) Distillation column heating element control panel.

As shown in Figure 0-1, a pyrolysis reactor with a feeding and temperature control flange is coupled to a distillation column to ensure the separation of the fractions obtained. The stirring system, consisting of a variable speed electric motor, a speed reducer, a sealing box and an agitator, ensures

proper mixing. The column, which has two sections, includes a distillation head [Figure 0-2(A)) equipped with a reflux divider to ensure an adequate reflux ratio. The heating elements are mounted on the column to simulate adiabatic operation [Figure 0-2(B)). The process temperature is set from the heating element control panel [Figure 0-2(C)). Three fractions - naphtha, diesel and the heavy fraction - are collected from the column. The collection rates of these fractions are set manually with the help of fine taps. The maximum capacity of the pyrolysis reactor is 3000 g of polymer waste.

3.2. Results and discussions

The yield of pyrolysis product (oil, coal and gas) was estimated based on the weight obtained for coal and oil at the end of the pyrolysis process. The yield in pyrolysis products of LDPE granules (1000 g) is shown in

Table 0-1.

Table 0-1. Products obtained from the pyrolysis of 1000 g LDPE (granules) (% wt.)

Catalyst			The product obtained			
Type	Quantity, g	Solid (w/o catalyst), %	Liquid, %			Gas, %
			Naphtha	Diesel	Heavy	
No cat.	-	18.3	10.8	15.9	16.6	38.3
Bentonite 20%	200	0	19.9	8.0	24.0	48.1
AMD1-20%	200	0	6.0	15.6	22.6	55.9
AMD1-10%	100	0	32.3	10.1	17.1	40.6
AMD1-5%	50	0.81	30.5	6.6	18.5	43.7
AMD4-5%	50	0	27.5	6.6	15.5	50.4

The pyrolysis experiments were carried out on plastic film waste from the sorting of household waste (IRIDEX). The waste contains not only plastic, but also paper, which absorbs a lot of water, the humidity being 23%; Part of this water is collected together with the liquid fraction, and another part goes together with the gases. The distribution of pyrolysis products, including liquid fractions, gases and solid residues were examined under different conditions. The results obtained from the pyrolysis of 900 g of this waste are presented in Table 0-2.

Table 0-2. Products obtained (g) from the pyrolysis of 900 g plastic film waste

Waste (g)	**Catalyst AMD1 (g)**	**Water (g)**	**Liquid fraction (g)**	**Gauze (g)**	**Solid residue (g)**
900	0	57.3	325.9	252	115.2
900	90	105.5	115.1	507.9	70

Pyrolysis experiments using polypropylene (PP) waste from the dismantling of automobiles produced three liquid fractions (light, diesel and heavy), a solid residue and a gaseous product. The influence of different catalytic systems on product distribution is summarized in Table 0-3.

Table 0-3. Distribution of products obtained from pyrolysis of polypropylene waste with different catalysts

Exp.	Temperature, °C		Results, %		
	Maximum liquid yield	Liquid depletion*	solid	liquid	Gas
AC_20	288	345	23.8	14.0	62.2
AC _10	298	347	24.2	16.3	59.5
AC _5	307	370	3.6	21.5	74.9
ACr_5	284	376	8.7	17.3	74.0
control	292	372	2.5	17.3	80.2
AMD4_5	287	367	1.8	12.5	85.7
AMD4+ AC _5+5	294	372	1.4	15.4	83.1

* at this temperature, the accumulation of liquid products ceases

The gas phase resulting from the catalytic pyrolysis of polypropylene waste was analyzed by gas chromatography. The analysis revealed a complex mixture dominated by hydrogen (H_2), methane (CH_4) and light hydrocarbons, along with smaller amounts of CO, CO_2 and C_5+ compounds. The presence and proportion of these components varied significantly depending on the catalyst used and its concentration, as shown in Table 0-4.

Table 0-4 Concentration of gas fraction obtained at polypropylene waste pyrolysis

Exp.	Concentration, vol.%													
	H_2	CO	CH_4	C_2H_6	CO_2	C_2H_4	C_3H_8	C_3H_6	i-C_4H_{10}	n-C_4H_{10}	1-C_4H_8	2-C_4H_8	C_5	C_6
AC_20	4.40	0.70	31.53	14.37	3.52	19.07	2.73	9.22	0.66	0.73	1.04	6.46	1.89	3.68
AC _10	4.31	0.27	21.03	18.78	1.03	20.22	3.69	12.37	0.55	0.86	1.35	7.12	3.70	4.72
AC _5	4.21	0.38	26.23	18.05	0.16	18.97	3.03	11.07	0.58	0.55	1.18	6.87	2.98	5.71
ACr	4.03	0.38	27.87	16.99	0.16	19.20	2.97	10.84	0.57	0.55	1.20	6.70	2.94	5.60
control	2.91	0.09	28.09	16.58	0.11	20.35	3.23	10.85	0.60	0.49	1.33	7.20	2.82	5.35
AMD4_5	4.3	0.0	29.8	17.5	0.2	18.1	3.3	9.1	0.7	0.6	1.1	5.7	2.9	6.6
AMD4 + AC _5+5	4.52	0.00	27.64	18.87	0.18	17.82	3.71	8.80	0.68	0.74	1.20	5.29	3.11	7.44

The distribution of hydrocarbon fractions in liquid products obtained from catalytic pyrolysis of polypropylene waste is presented in

Table 0-5. The composition has been classified into three distillation ranges: light hydrocarbons (C_4-C_{13}), diesel (C_{14}-C_{19}) and heavy hydrocarbons (C_{20}+).

Table 0-5. Composition of liquid fraction in pyrolysis of polypropylene waste

Exp.	C_4 - C_{13}, wt.%	C_{14} - C_{19}, wt.%	C_{20}+, wt.%
A_20	83.76	13.13	3.06
AC_10	79.84	16.36	3.76
AC_5	82.03	14.70	3.27
ACr_5	80.96	15.26	3.78
Control	83.96	12.79	3.24
AMD4_5	81.32	13.68	4.99
AMD4+ AC_5+5	88.72	9.65	1.62

3.5.Partial conclusions on LDPE and PP pyrolysis:

Experimental studies carried out on catalytic pyrolysis of low-density polyethylene (LDPE) and polypropylene (PP) waste have highlighted both common characteristics and significant differences in thermochemical behavior and product distribution. In the case of LDPE, the use of catalysts such as bentonite and zeolite led to substantial changes in the composition of liquid and gaseous fractions, by increasing the light fraction from 22% to over 60% and by doubling the amount of gas generated in the PE waste pyrolysis process. Also, the catalytic effect led to a visible aromatization of the products, with concentrations of aromatic hydrocarbons that reached up to 21.2%, which can contribute to increasing the energy and chemical value of the liquid fractions.

Similarly, the catalytic pyrolysis of polypropylene demonstrated a high potential for conversion into alternative fuels, in particular due to the production of gases with a calorific value of more than 72,000 kJ/m³ and the high concentration of C4–C13 hydrocarbons in the liquid phase (up to 88.72% by weight). The use of activated carbon combined with the AMD4 catalyst allowed to optimize the composition of the products and increase energy efficiency, the results indicating the feasibility of using the gases produced directly to power the motor generators and produce electricity. Overall, this research demonstrates that the application of catalytic pyrolysis to polyolefin waste, either LDPE or PP, is a viable advanced recycling strategy, with major benefits for the circular economy, pollution reduction and sustainable energy recovery of plastics.

CHAPTER IV

MICROWAVE-ASSISTED PYROLYSIS OF PLASTIC WASTE WITH PE AND PP CONTENT, RESULTING FROM THE DISMANTLING OF VEHICLES: HYDROGEN PRODUCTION AND ENERGY RECOVERY

4.1.Materials and methods

The reactor is loaded with layers of plastic, activated carbon, and silicon carbide, then placed in a single-mode microwave cavity. Microwave heating (max. 660°C for 10 minutes) is monitored in real time. Gaseous and liquid products are collected and analyzed, while solid residues are weighed post-reaction. The yields in the fractions obtained and the calorific value of the gas are calculated to evaluate the efficiency of the process.

Silicon carbide (SiC) (Green Silicon Carbide, 2-4 mm, Sinabuddy Mineral, China), activated carbon (AC) (Coconut Shell 6*12 mesh, Legend Inc. USA, polypropylene (PP) granules (referred to as PP_T) and polyethylene granules (LDPE MI 2) (referred to as PE_T) were purchased from Romcolor, Copăceni, Romania [3]. In addition, polypropylene obtained from car dismantling (referred to as PP_W) and two types of mixed waste – composed of polypropylene and polyethylene – collected from automotive components obtained from SC Pieseauto Dez SRL, Constanta, Romania (designated as PP+PE_W1 and PP+PE_W2 respectively), were used as representative raw materials for plastic waste.

Activated carbon was used as a catalytic agent in different mass concentrations, while silicon carbide (SiC) served as a microwave susceptor to allow efficient heating during pyrolysis. Activated carbon acted as a solid-phase catalyst to promote the thermal decomposition of plastic waste, while SiC primarily absorbed microwave radiation and quickly transferred heat to the reaction system due to its dielectric properties.

4.3.Microwave Assisted Pyrolysis Plant Setup

The pyrolysis experiments were carried out in a custom quartz tube reactor (17 mm inner diameter and 20 mm outside) placed in a single-mode microwave cavity operating at a maximum power of 700 W; frequency: 2.45 GHz; made by Sairem France). The installation configuration is shown in the Figure 0-1.

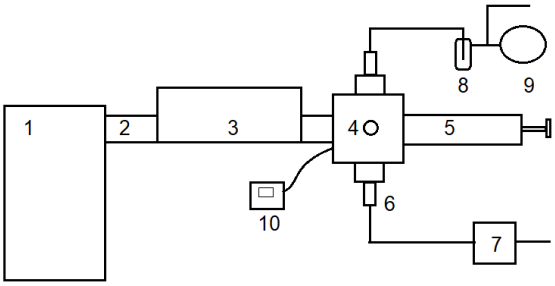
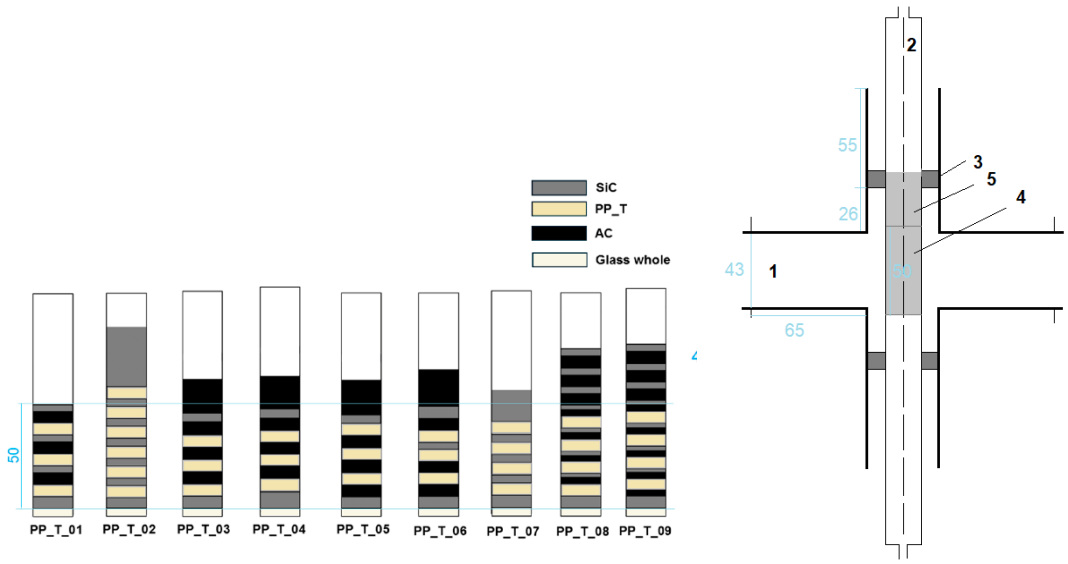


Figure 0-1. Microwave catalytic pyrolysis plant. 1 – GMP 30K SM 56T400 FST 3 IR microwave generator (Sairem – France); 2 – WR340 standard waveguide (Sairem France); 3 – 2.45 GHz AI 4S A 2450/340 automatic tuner (Sairem – France); 4 – Single-mode cavity (Sairem – France); 5 – Sliding Short Circuit PCCMWR340L130PVMR1PE (Sairem – France); 6 – Quartz reactor; 7 – Digital mass flow control for nitrogen – Smart-Trak® 50 series (Sierra – USA); 8 – Liquid fraction collection vessel; 9 – 1 liter Tedlar bag (CEL Scientific Corporation – USA) for the collection of the gaseous fraction; 10 – Micro-epsilon infrared sensor (Messtechnik – Germany).

4.4.Results and discussions

In the single-mode applicator, microwave energy absorption is achieved only in the presence of silicon carbide. For uniform heating of plastic granules, it is necessary to alternate between plastic and silicon carbide layers. To evaluate the influence of catalyst loading and layering on polypropylene (PP) pyrolysis, nine different experimental setups were designed, labeled PP_T_01 to PP_T_09. These configurations varied in the quantity and distribution of silicon carbide (SiC) and activated carbon (CA), which were alternately stratified with the PP feedstock inside the reactor (Figure 0-2and Figure 0-3A). The visual representation of Figure 0-2show light gray layers as PP, dark gray as SiC, and black as CA.



(a) **(b)**

Figure 0-2. Schematic Representation of Layered Configurations in the Quartz Reactor: (a)layered configurations; (b) Quartz reactor in the single-mode cavity: 1 – Single-mode cavity; 2 – quartz reactor; 3 – Teflon rings covered with aluminum foil for centering the reactor; 4 – Filling the reactor, in the active area; 5 – Additional reactor filling. The dimensions of the single-mode cavity and quartz reactor are shown in mm.



A B

Figure 0-3. (A) Image of SiC, CA and feedstock layers in quartz reactor; (B) Single-mode cavity and 2.45 GHz automatic tuner.

Each setup was pyrolysis at 650°C for 10 minutes in a nitrogen atmosphere, using a single-mode microwave reactor and microwave generator power of up to 600W. The corresponding distributions of gaseous, liquid and solid products have been recorded and are summarized in Table 0-1.

Table 0-1. Conversion of raw material and hydrogen concentration for each reactor configuration using technical polypropylene raw material.

Configuration	Quantity (g)			Content (%)		Conversion, %	H_2,vol.%
	Raw material	AC	SiC	SiC	CA		
PP_T_01	3	3	5	45.45	27.27	55	7.25
PP_T_02	6	0	15	71.43	0.00	66	16.19
PP_T_03	3	6	4	30.77	46.15	62.3	19.75
PP_T_04	3	6	5	35.71	42.86	9.5	10.41
PP_T_05	3	6	4	30.77	46.15	40.3	19.27
PP_T_06	3	6	5	35.71	42.86	57	25.37
PP_T_07	4	0	8	66.67	0.00	93.50	22.74
PP_T_08	4	5	7	43.75	31.25	92.75	13.87
PP_T_09	4	5.5	8	45.71	31.43	90.3	45.23

Due to its superior performance, the PP_T_09 configuration was selected as the reference configuration for subsequent experiments involving different sources of polypropylene and polyethylene waste. Subsequently, this experiment will be noted as PP_T.

The type of plastic raw material used in the pyrolysis process significantly influenced both the gas yield and the composition of the gas fraction, especially the hydrogen content. The experiments were carried out using polypropylene granules (PP_T), polyethylene granules (PE_T) and plastic waste

from automotive dismantling operations, which included both PP waste (PP_W) and 2 types of PP+PE waste mixtures (PP+PE_W1 and PP+PE_W2). The results are presented in Table 0-2.

Table 0-2. Distribution of products obtained by conventional or microwave-assisted pyrolysis of various raw materials (polypropylene and polyethylene)

Exp.	Results, wt.%				Conversion %	Efficiency of H_2 g/g	Yield H_2, mmol/g plastic
	Unreacted	solid	liquid	gas			
PE_T	7±0.15	12.1±0.6	9.95±1.1	77.9±1.5	93.0±0.4	0.00	14.3±0.6
PP_T	9.75±0.3	6.65±1.2	11.91±1.3	81.44±1.5	90.3±0.3	16.19	32.0±0.3
PP_W	ID: 5±0.05	10.72±0.7	11.39±1.5	77.89±1.8	95.0±0.2	19.75	9.5±2.4
PP+PE_W1	1±0.2	22.32±2.1	10.8±1.2	66.7±1.7	99.0±0.5	10.41	34.4±0.4
PP+PE_W2	3.17±0.7	4.48±0.03	8.5±2	87.08±1	96.8±0.6	19.27	6.2±0.1
PP_W_conv_1	3.6±0.1	3.46±0.5	8.92±0.8	87.62±1.9	96.3±0.3	25.37	4.77±1.4
PP_W_conv_2	0.2±0.05	0.1±0.02	14.3±0.7	85.6±2.1	99±0.2	22.74	2.6±1.2
PP_W2	16.5±0.8	4.4±0.6	10.4±0.7	85.2±1.8	83.3±0.5	37.2±0.5	26.5±0.8

The composition of the gas fraction resulting from microwave-assisted pyrolysis varied significantly depending on the type and origin of the plastic raw material (Table 0-3). Primary gaseous products included hydrogen (H_2), methane (CH_4), ethane (C_2H_6), ethylene (C_2H_4), propane (C_3H_8), propylene (C_3H_6), butane-butene and small fractions of carbon oxides (CO, CO_2), as well as heavier hydrocarbons (C_5-C_6).

Table 0-3 Composition of the gas fraction obtained by conventional and microwave-assisted pyrolysis of different types of plastics

Exp.	Concentration, vol.%													
	H_2	CO	CH_4	C_2H_6	CO_2	C_2H_4	C_3H_8	C_3H_6	i-C_4H_{10}	n-C_4H_{10}	1-C_4H_8	2-C_4H_8	C_5	C_{6+}
PE_T	36.63	2.11	22.76	5.42	0.71	18.39	1.94	3.93	0.00	1.00	2.04	1.92	0.57	2.59
PP_T	45.23	1.04	43.28	8.61	1.51	0.03	0.03	0.15	0.01	0.00	0.00	0.07	0.02	0.02
PP_W	46.50	0.78	16.52	4.25	4.09	15.53	1.89	5.41	0.22	0.15	0.59	2.06	0.48	1.52
PP+PE_W1	75.35	0.16	0.29	7.07	0.35	0.68	1.21	3.35	0.36	0.29	0.30	2.68	1.51	6.41
PP+PE_W2	20.05	0.68	23.32	8.25	2.50	19.64	1.79	7.96	0.95	0.58	1.16	4.92	2.25	5.93
PP_W_conv_1	16.51	0.35	26.62	9.65	3.24	13.34	2.31	7.65	1.38	1.48	1.11	5.90	3.29	7.16
PP_W_conv_2	9.32	0.00	30.00	10.69	0.00	21.00	2.16	9.87	0.30	0.43	1.38	6.55	1.69	6.61
PP_W2	46.16	2.37	27.07	2.33	0.54	14.32	1.2	2.43	0.16	0.07	0.31	1.52	0.37	1.15

The gross heating value (GHV) and CO_2 emissions of the gaseous products resulting from pyrolysis varied significantly depending on the type of plastic raw material and the heating method applied, which are presented in the Table 0-4.

Table 0-4. Gross calorific value and CO2 emissions of gaous fractions obtained by microwave-assisted and conventional pyrolysis of different types of plastics

Exp	GHV mixture,		CO_2 emissions	Specific CO_2 emissions
	kJ/m^3	kJ/kg	L CO_2/L gas mixture	kg CO_2/kJ
PE_T	44398.25	49750.55	1.28	5.68E-05
PP_T	27959.21	54320.32	0.63	4.40E-05
PP_W	37577.78	47625.47	1.36	5.82E-05
PP+PE_W1	36016.20	55087.35	0.90	4.92E-05
PP+PE_W2	59826.72	46941.68	1.86	6.12E-05
PP_W_conv_01	64272.68	46313.50	2.02	6.17E-05
PP_W_conv_02	66926.83	48457.28	2.12	6.23E-05
PP_W2	34509.63	52137.78	0.91	5.16E-5

From an environmental perspective, specific CO_2 emissions from pyrolysis gases were generally lower or comparable to commercial fossil fuels. The cleanest composition was observed in PP_T (4.40×10^{-5} kg CO_2/kJ), significantly lower than propane (5.96×10^{-5}), natural gas (5.01×10^{-5}) and significantly better than kerosene (6.94×10^{-5}) or diesel (7.03×10^{-5}). Even the most carbon-intensive sample, PP_W_conv_02, remained within a competitive range of 6.23×10^{-5} kg CO_2/kJ, between the propane and kerosene range.

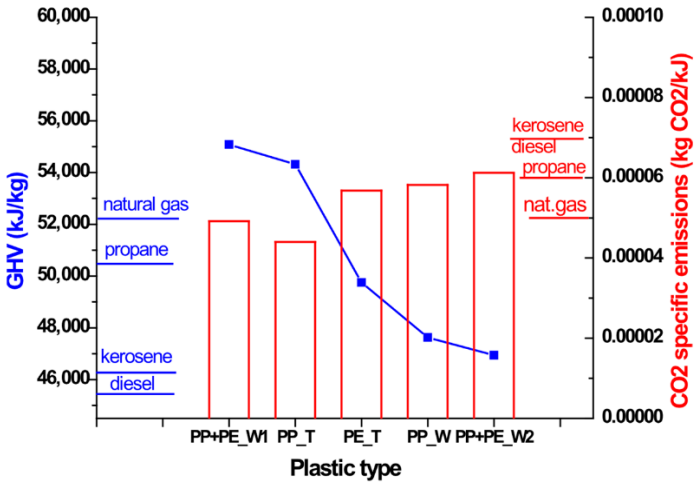


Figure 0-4. Calorific value (GHV) and specific CO_2 emissions of the gaseous fraction obtained from microwave-assisted pyrolysis of plastic compared to conventional fuels

4.5.CONCLUSIONS

Microwave-assisted pyrolysis leads to obtaining a preponderance of gaseous fraction (67-87 % gr.) and solid fraction (4.5-22%) and mimicry of the liquid fraction (7-14%).

The gas obtained from the pyrolysis of plastic waste, especially from polypropylene, has shown significant potential as an alternative fuel. The gas produced exhibits a high energy density, making it a promising sustainable energy source. Additionally, this approach could help reduce carbon emissions by aligning with global efforts to minimize the environmental impact of waste.

Microwave-assisted pyrolysis shows considerable promise in producing cleaner gas compositions with a very good value for GHV (kJ/kg). The use of microwaves increases selectivity towards lighter hydrocarbons, which are very valuable for refining fuels, thus providing a potential avenue for the production of more refined fuels.

The study also highlights the effectiveness of microwave-assisted pyrolysis in hydrogen production. This method results in a higher hydrogen yield (up to 34.4 mmol H2/g plastic), with improved efficiency compared to conventional pyrolysis. The increase in hydrogen production highlights the potential of microwave energy in decomposing polypropylene and promoting the formation of hydrogen-rich gases, which are of great interest for future energy applications.

CHAPTER V

THERMOCATALYTIC PYROLYSIS OF LIGNOSULFONATE

5.1.Experimental part

The pyrolysis reactor used in lignosulfonate processing has a horizontal configuration, made of stainless steel, with an inner diameter of Ø 25.4 mm and a length of 250 mm. In the reactor there is a thermocouple for measuring the internal temperature. For heating, an electric oven with digital temperature control is used.

Before each experiment, inert gas (nitrogen) is purged into the reactor to remove oxygen. Figure 0-1 It shows a schematic representation of the pyrolysis reactor, where the inert gas flow rate is regulated using a mechanical valve and a flow meter. The resulting pyrolysis products are cooled in a heat exchanger and subsequently enter a gas-liquid separator. The liquid fraction is collected, prepared and subjected to analysis by gas chromatography (GC-MS).

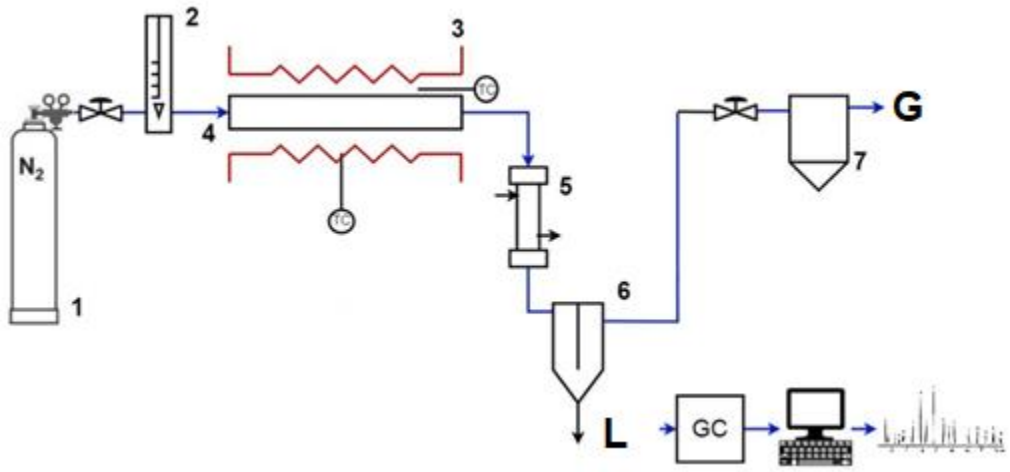


Figure 0-1. Ligno-sulfonate pyrolysis plant. 1-gas cylinder, 2-flow meter, 3- electric heating furnace, 4- pyrolysis reactor, 5-heat exchanger (cooler), 6- gas-liquid separator, 7-gas tank, TC- thermocouple and heat display system, GC- gas chromatograph

Pyrolysis reactions were performed at 500°C for 4 hours, the reactor being heated to operating temperature with a heating rate of 20°C/min. The mixture of conditioned lignosulfonate and catalyst was ground and subsequently fed into the reactor in ceramic nacelles. The concentration of the catalyst was maintained at 2% (mass) relative to the mass of lignosulfonate.

5.2.Results and discussions

Thermogravimetric analysis shows the thermal behavior of the sample, revealing distinct patterns of mass loss at different temperature ranges. Initially, a mass loss of 6.05% up to 140ºC is observed, which can be attributed to the presence of water and volatile components in the sample. Subsequently, in the temperature range of 180-550ºC, the mass loss curve shows three distinct peaks at 218.3ºC, 270.4ºC and 395.0ºC. These peaks correspond to the decomposition of hemicellulose and lignin [4].

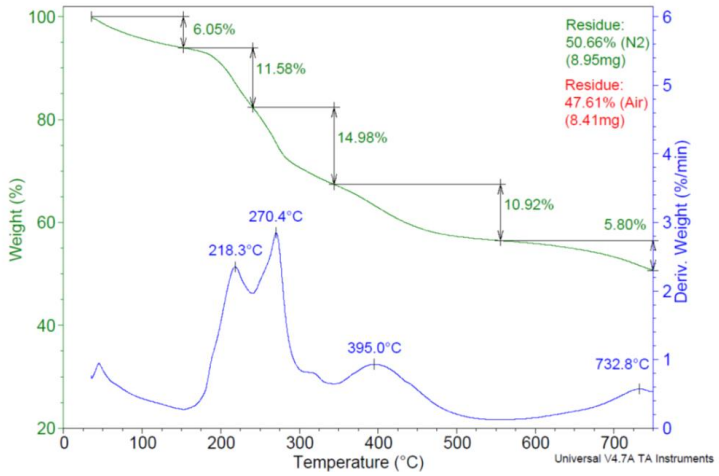


Figure 0-2. Thermogravimetric analysis of lignosulfonate

The structural characteristics of the catalysts were obtained using nitrogen porosimetry and are presented in Table 0-1. Prior to analysis, catalyst samples were subjected to a vacuum degassing process for 4 hours at 160ºC. The resulting catalysts are mesoporous materials with pore diameters ranging from 4 nm to 10 nm.

Table 0-1. Characteristics of catalysts

No.	Catalyst	Specific area, m^2/g	Total pore volume, cm^3/g	Average pore diameter, nm
2	Mo/Al_2O_3	157.3	0.244	6.207
3	$NiMo/Al_2O_3$	236.8	0.2839	4.795

The analysis of bio-oil obtained by thermal pyrolysis of conditioned lignosulfonate is presented in Table 0-2 and Figure 0-3. The GC-MS chromatogram shows a multitude of peaks. The composition of the liquid phase indicates the presence of oxygenated compounds with aromatic structure - substituted phenols and phenols, linear and cyclic oxygenated aliphatic compounds, including carbonyl compounds, acids and alcohols.

Table 0-2. GC-MS analysis of lignosulfonate bio-oil obtained without catalyst

No.	Retention Time	Name of compounds
1	5.335	2-butanone, 3-hydroxy-
2	5.551	Acetic anhydride
3	6.460	2-Hepten-1-ol, (E)-
4	7.322	Acetic acid
5	7.561	2-Propanone, 1-(acetyloxy)-
6	9.484	Butanoic acid
7	9.907	Furfuril alcohol
8	11.415	1,3-cyclopentanedione, 2,4-dimethyl-
9	11.495	Cyclohexanone, 2-acetyl-
10	11.822	2-cyclopenten-1-one, 2-hydroxy-3-methyl-
11	12.029	Cyclohexanone, 2-acetyl-
12	12.173	Phenol, 2-methoxy-
13	13.649	Phenol
14	16.146	Phenol, 2,6-dimethoxy-
15	19.473	2-Propanone, 1-(4-hydroxy-3-methoxyphenyl)-
16	23.646	Desaspidinol

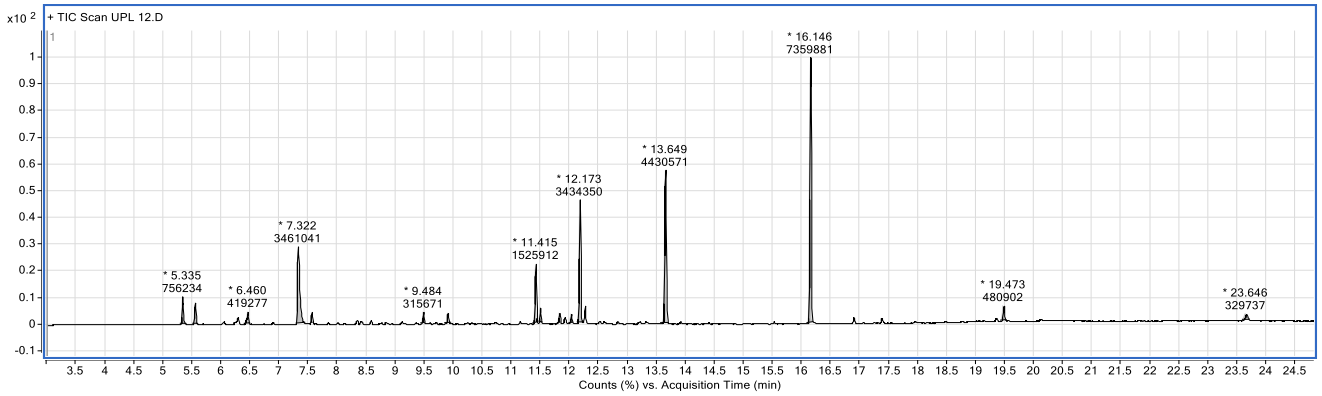


Figure 0-3. Chromatogram of lignosulfonate bio-oil obtained from pyrolysis without catalyst

The porosimetric analysis of the biochar obtained showed that the pyrolysis of the residues produces carbonaceous materials characterized by a specific surface area and a reduced pore volume, while retaining adsorption capacities for the chemical compounds. In contrast, biochar that possesses an increased surface area is considered more porous, making it suitable for applications such as water treatment and environmental remediation [5, 6].

GC-MS analysis of bio-oil produced using Mo/Al_2O_3 is presented in Table 0-3 and Figure 0-4. The homogenization of the catalyst and the conditioned lignosulfonate particles was done by grinding them together in the ball mill.

Table 0-3. GC-MS analysis of bio-oil obtained by pyrolysis in the presence of Mo/Al catalyst$_{2O3}$

No.	Retention Time	Name of compounds
1	4.745	2-butanone, 3-hydroxy-
2	4.929	2-Propanone, 1-hydroxy-
3	5.567	Furan, 3-methyl-
4	6.253	Acetic acid
5	6.684	1,2-Ethanediol, diacetate
6	7.474	Propanoic acid
7	7.681	2-cyclopenten-1-one, 2,3-dimethyl-
8	8.511	Butanoic acid
9	10.434	1,3-cyclopentanedione, 2,4-dimethyl-
10	11.192	Phenol, 2-methoxy-
11	12.644	Phenol
12	12.915	Phenol, 4-ethyl-2-methoxy-
13	15.174	Phenol, 2,6-dimethoxy-
14	15.892	Phenol, 4-methoxy-3-(methoxymethyl)-
15	16.370	Benzene, 1,2,3-trimethoxy-5-methyl-
16	18.477	2-Propanone, 1-(4-hydroxy-3-methoxyphenyl)-

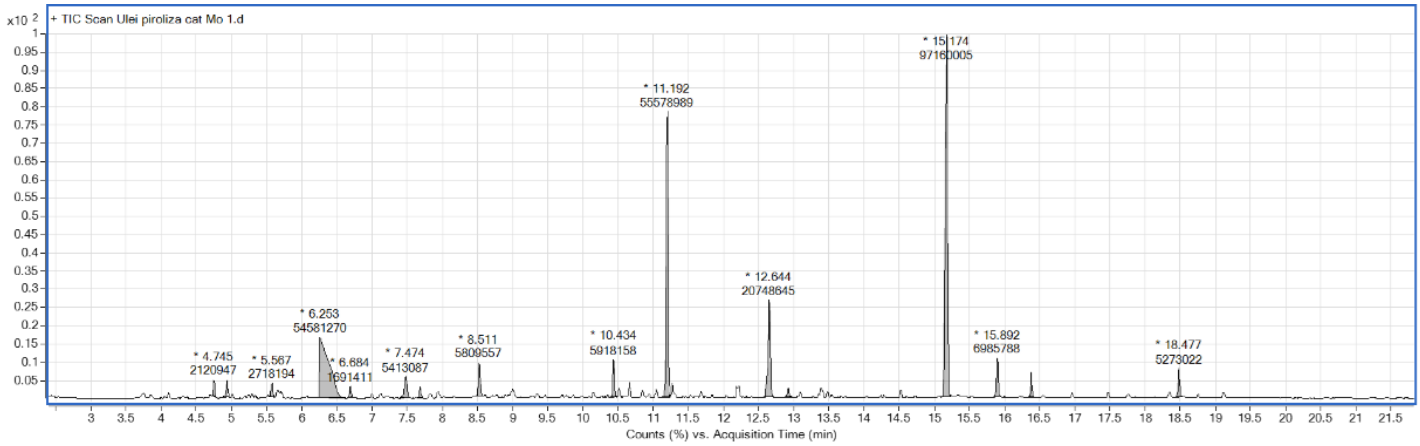


Figure 0-4. Chromatogram of the bio-oil obtained from the pyrolysis of ligno-sulfonate in the presence of the Mo/Al2O3 catalyst

From the GC-MS analysis data, it was found that the liquid phase includes oxygenated compounds with aromatic structures, phenolic compounds, furans, substituted benzene, and linear/cyclic aliphatic oxygenated compounds - carbonyl compounds and organic acids. It was observed that the presence of a Mo-based catalyst during pyrolysis resulted in a higher concentration of organic acids and a higher concentration of furan compounds compared to a Ni-Mo-based catalyst. The results obtained support the findings reported in the literature [7]. Molybdenum-based catalysts exhibit a C-C bond cleavage in the side chains, resulting in significant production of carboxylic compounds [8, 9]. The addition of nickel, in turn, suppresses the reaction between aromatic substances and other oxygenated substances, producing a variety of alkylated aromatic compounds [10, 11].

Table 0-4. Overview of the results obtained by pyrolysis of lignosulfonate

Identified compounds	Without catalyst	Mo Catalyst, %	Ni-Mo Catalyst, %
Furan compounds	1.15	0.99	0.48
Aliphatic ketone	15.77	6.47	7.77
Organic acids	15.42	24.03	17.29
Phenolic compounds	62.17	66.46	68.76
Other compounds	5.49	2.06	5.70

5.3.CONCLUSIONS

This study presents the conditioning of lignosulfonate used in the production of cellulose from lignocellulosic biomass and the thermal and catalytic pyrolysis of conditioned lignosulfonate, focusing on the production and analysis of bio-oil and biochar. The obtained results provide valuable information about the impact of lignosulfonate catalytic pyrolysis and its potential applications. The use of molybdenum and nickel-molybdenum catalysts showed distinctive effects on bio-oil yield, chemical composition and product distribution. GC-MS analysis revealed various compounds, including oxygenated species such as phenols, furans, aliphatic compounds, and carbonyl compounds in the liquid phase of bio-oil. The porosimetric analysis of the biochar resulting from pyrolysis shows

the role of the catalyst in determining the porosity of biochar. The presence of the molybdenum catalyst leads to biochar with a reduced specific surface area, while the use of a nickel-molybdenum catalyst improves it substantially. The effects of using catalytic pyrolysis compared to non-catalytic pyrolysis for the recycling of lignosulfonate residues and for the characterization of materials obtained from this process are presented.

GENERAL CONCLUSIONS

In

Table **0-5** and Table 0-6 are centralized results for pyrolysis of polyolefins and lignosulfonate waste are presented.

Regarding the pyrolysis of polyolefins, it can be seen that two distinct research directions have been addressed:

- Conventional pyrolysis, carried out in a pilot plant with a capacity of max. 3 kg and equipped with a distillation column. In this installation, the aim was to improve the quality of the liquid fraction by using various catalysts. It was possible both to increase the share of light fractions and to improve their quality (increasing the share of hydrocarbons with branched or cyclic chains);
- Microwave-assisted pyrolysis was carried out in a laboratory facility. The use of a single-mode applicator has made it possible to transform the efficiency of plastic, especially into gaseous compounds in which hydrogen predominates. In this context, it becomes much simpler to use the fractions obtained with minimal CO_2 emissions to energy.

In the pyrolysis processes studied, both technical polyolefins and polyolefin waste (polyethylene films separated from household waste and PE and PP plastic separated from waste from vehicle dismantling) were used, thus paving the way for the development of industrial processes for recycling plastic waste.

Table 0-5. Centralized polyolefin pyrolysis results

Polyolefin pyrolysis	Conventional pyrolysis	Microwave assisted pyrolysis
Installation type	Pilot plant (1-3 kg of plastic waste) equipped with distillation column	Laboratory installation, single-mode cavity, capacity 10-15 g of which 3-4 grams of plastic waste
Polyolefin type studied	• PE and PP technical granules; • PE waste film type; • PP waste from car dismantling.	• PE and PP technical granules; • PP waste from car dismantling; • PP+PE waste from car dismantling.
Waste characterization	-	Waste from car dismantling was characterized by: • TGA/DTG analyses; • DSC analyses; • FTIR analyses; • XRD analysis.
Catalysts used	• Clinoptilolite; • modified zeolite (AMD 4); • Activated carbon.	• Activated carbon; • SiC type susceptors.
Reactor type	With mixing of the catalyst into the plastic melt.	Tubular, with the catalyst; The Susceptor and the Plastic Arranged in Layers
What was followed	Increasing the quality of the liquid fraction by: • increasing the share of fraction C_5-C_{13}; • increasing the share of branched-chain and aromatic hydrocarbons; Catalyst recirculation; Comparison of non-catalytic and catalytic process.	Obtaining an optimal configuration of the layers for the most efficient heating; Increasing the share of the gaseous fraction (>80% by mass); Increasing the quality of the gaseous fraction by increasing the hydrogen content (efficiency of obtaining hydrogen). Catalyst recirculation Comparison between the results obtained by classical pyrolysis and microwave-assisted pyrolysis (using the same tubular reactor).

Regarding the pyrolysis of lignosulfonate waste, the use of catalysts to improve the quality of the liquid fraction obtained was pursued. It is known that when pyrolysis of biomass waste, the liquid fraction has a very complex composition, which makes it difficult to process. The use of Mo and Ni/Mo catalysts on alumina support modified both the composition of the liquid fraction (the concentrations of organic acid-type compounds increased significantly) and the properties of the biochar obtained.

Table 0-6. Results of Waste Ligosulfonate Pyrolysis Process

Installation type	Laboratory plant with electric heating, tubular reactor, static, gram capacity
Raw material type	Dry technical lignosulfonate, from industrial processes to obtain cellulose
Waste characterization	TGA analyses for thermal behavior; GC-MS for chemical structure; DLS for particle size assessment
Catalysts used	Mo/Al_2O_3; $NiMo/Al_2O_3$
Reactor type	Horizontal configuration, with series operation, made of stainless steel, with an inner diameter of Ø 25.4 mm and a length of 250 mm.
What was followed	Obtaining liquid fractions with high content of aromatic and phenolic compounds, reducing the solid fraction by more complete conversion

DEVELOPMENT PROSPECTS

Based on the results obtained in this research, several directions of applied and scientific development of polyolefin pyrolysis processes can be identified, with a focus on increasing efficiency, sustainability and technological scalability:

- Conducting further research at laboratory and pilot scale on the separation of catalysts from the carbon residue and the possibility of their reuse. One of the critical aspects in catalytic pyrolysis processes is the contamination of catalysts with solid carbon fractions (char), future studies may aim to optimize the separation and regeneration procedures of catalytic materials. Methods of washing, controlled thermal activation or chemical treatment of recovered catalysts can be tested in order to assess the degree of maintenance of their activity in repeated pyrolysis cycles. This research will help reduce operational costs and increase the sustainability of the process.
- The carbon residue obtained after pyrolysis of polyolefins contains a significant fraction of fixed carbon and can be a source of value if properly processed. Prospects include the use of this material as an adsorbent in gas or wastewater purification processes, as a carrier material for catalysts or as a potential solid fuel with specific energy characteristics. Future research can evaluate the physicochemical properties of the residue (specific surface, porosity, elemental composition) and identify applications with favorable economic impact.
- A prospective objective is to contribute to the realization of conventional and microwave-assisted industrial pyrolysis plants, starting from the data obtained in the experimental studies. The integration of microwave and conventional heating systems, together with the optimization of catalytic configurations, can lead to the creation of energy-efficient and economical installations, capable of processing large volumes of plastic waste. In this context, it will be possible to move on to the elaboration of technical-economic documentation, the development of pilot-sized

prototypes and, subsequently, the implementation on an industrial scale, thus contributing to the development of sustainable advanced recycling technologies.

DISSEMINATION OF RESULTS

Published articles

1. **Psenovschi, G.**, Calinescu, I., Fiti, A., Chisega-Negrila, C. G., Ionascu, S. L., & Barbes, L. (2025). Microwave-Assisted Pyrolysis of Polyethylene and Polypropylene from End-of-Life Vehicles: Hydrogen Production and Energy Valorization. *Sustainability*, *17*(13), 6196. ; https://doi.org/10.3390/su17136196; WOS:001527569400001 (F.I.=3.3)

2. Calinescu I, **Psenovschi G**, Cojocaru M, Chisega-Negrila CG, Albulescu C, Brebu M, Trifan A, Ignat ND, Chipurici P. Catalytic pyrolysis of low-density polyethylene waste. *Sustainability*. 2024; 16(16):6788; https://doi.org/10.3390/su16166788; WOS:001307534200001 (F.I.=3.3)

3. Gavrila, Adina I., Anamaria Vartolomei, Ioan Calinescu, Mircea Vinatoru, Oana C. Parvulescu, **Grigore Psenovschi**, Petre Chipurici, and Adrian Trifan. "Ultrasound-assisted alkaline Pretreatment of biomass to enhance the extraction yield of valuable chemicals." *Agronomy* 14, no. 5 (2024): 903; https://doi.org/10.3390/agronomy14050903; WOS:001233105800001 (F.I.=3.4)

4. **Pşenovschi, G**., Calinescu*, I., Fiti, A., Chisega-Negrila, C-G, "Catalytic pyrolysis of polypropylene waste**", accepted for publication** U.P.B SCI. BULL., SERIES B,

5. Vasilievici, G., Ghimiș, S. B., **Pșenovschi, G.**, & Sîrbu, M. (2023). "Thermo-Catalytic Pyrolysis of Lignosulfonate from the Sulfite Process". *Studia Universitatis Babes-Bolyai, Chemia*, *68*(4). (F.I.=0.5)

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2. Mirt L., Vlaicu A., Ciltea-Udrescu M., **Psenovschi G.**, Vasilievici G. "Catalytic pyrolysis of reed for bio-oil and biochar production". 19th International Symposium "Priorities of Chemistry for a Sustainable Development" PRIOCHEM, Bucharest, Romania, 11-13 October 2023.
3. **Grigore Psenovschi**, Ioan Călinescu, Petre Chipurici, Mihaela Cojocaru, Ciprian Chisega Negrilă, "Fuels from Waste Plastics by Pyrolysis", SICHEM 2024.
4. **Grigore Psenovschi** , Petre Chipurici, Mihaela Cojocaru, Ciprian Chisega Negrila, Anca Paulenco, Ioan Calinescu "Conversion of Waste Plastics to Fuels Through Pyrolysis" 20th International Symposium "Priorities of Chemistry for a Sustainable Development" PRIOCHEM, Bucharest, Romania, 2024.

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