Universitatea "Lucian Blaga" din Sibiu

Interdisciplinary PhD school

PhD domain: Industrial Engineering

PhD THESYS

RESEARCH REGARDING THE ENERGY GENERATION FROM SOLID WASTE AND PLASTIC MATERIALS THROUGH THE DEVELOPMENT OF ALTERNATIVE FUELS

PhD student:

Chemist Marius Constantinescu

Scientific advisor:

Prof. univ. dr. Simona OANCEA

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List of abbreviations

A - ash

AAS - atomic absorption spectroscopy

AR4 - fourth evaluation report

AW - animal waste

BS - beech sawdust biomass

BW - biomass vegetable waste (apricot kernels, grape waste)

C - carbon

C_6H_5-COOH - benzoic acid

-CH_2 - methylene

-CH_3 - methyl

CH_4 - methane

CLU - light liquid fuel

CO - carbon monoxide

CO_2 - carbon dioxide

CO_x - carbon oxides

EA - elementary analysis

ESS - innovative solid energy sort

ETBE - third butyl ether

EF - emission factor

FID - detector with ionization flame

FTIR - Fourier transform infrared spectroscopy

GC - gas chromatography

GC-FID - gas chromatography with ionization flame detector

GC-MS - gas chromatography coupled with mass spectrometry

GC-TCD - gas chromatography with thermal conductivity detector

GHG - greenhouse gases

GWP - global warming potential

H - hydrogen

H_2 - hydrogen

H_2O - water

H_2S - hydrogen sulfide

HDPE - high density polyethylene

HFO - heavy fuel

HPLC - high performance liquid chromatography

L - lignite

LDPE - low density polyethylene

LFO - light fuel

LPG - liquefied petroleum gas

MBM - meat and bone meal

MCM 41 - mesoporous silica

N - nitrogen

NH_3 - ammonia

NO - nitrogen monoxide

NO_2 - nitrogen dioxide

NO_x - nitrogen oxides

O - oxygen

OW - olive waste

PAH - polycyclic aromatic hydrocarbons

PE - polyethylene

PET - polyethylene terephthalate

PM1.0 - particles in suspension fraction 1 micron

PM10 - particles in suspension fraction 10 microns

PM2.5 - particles in suspension fraction 2.5 microns

PP - polypropylene

PPG - pyrolysis gas (gas from pyrolysis of plastics)

PPO - pyrolysis oil (plastic pyrolysis oil)

PPPW - experimental installation for pyrolysis of plastic samples

PPW - wax / pyrolysis residue (plastic pyrolysis wax)

PS - polystyrene

PVC - polyvinyl chloride

Q_i - superior calorific value

Q_s - higher calorific value

S - sulfur

SAW - pellets obtained from sewage sludge and animal waste

SBA-15 - mesoporous silica

SEM - scanning electron microscopy

SFBR - single fluidized bed reactor

SM 5A - molecular sieve

SO_2 - sulfur dioxide

SOW - pellets based on sewage sludge and olive waste

SO_x - sulfur oxides

SS - sewage sludge

SSMS - metal blocks of sandwich type catalyst support

T - temperature

t - time

TCD - thermal conductivity detector

TEC - equivalent total toxicity concentration

TEF - toxic equivalence factor

TGA - thermal gravimetric analysis

TSP - total powder in suspension

U.E. - European Union

V - volatile matter

V_2O_5 - vanadium pentoxide

W - humidity

WHO - World Health Organization

WWTP - Wastewater Treatment Plant

Z - slag

ZSM-5 - zeolite

PhD thesis summary

1. Introduction

In recent years, through population growth, urbanization, accelerated industrialization, the amount of waste has normally increased exponentially. An essential element in sustainable development policy in the U.E. it is the exploration and efficient exploitation of resources that can generate raw materials for the development of renewable energy, a fact legislated by specific directives.

The sustainability of the research in this doctoral thesis is based on the need to create products - solid, liquid and gaseous - with high energy potential, innovative, relatively inexpensive, with a low footprint of NO_x, CO_2, SO_x emissions, starting from waste. Renewable energy technologies offer investment opportunities and represent workforce potential and can be the premise for energy security. At the moment, the use of waste for energy purposes is the new "El Dorado" in Europe and also in the USA, which means the huge potential in terms of financial benefits. The sustainability of resources is based on the exploration of existing local alternative sources, exploitable for the development of new fuels, alternatives to existing fossils. Through the solution adopted, for the use of new raw materials, respectively special solid waste: *(i)* sewage sludge from municipal wastewater treatment plants (SS); *(ii)* biomass / waste (pips, sawdust, grit, straw) (BS; BW); *(iii)* bone meal / mechanically boned meat / animal waste (MBM); *(iv)* slag - from the energy exploitation of coal, respectively lignite (Z); *(v)* plastic waste; having a mandatory feature - local traceability, it creates premises to achieve low costs for final products.

The main objective was to find sustainable and perfectly feasible processes for the return to the circular economy of the above-mentioned waste as raw materials. The exchange, initially utopian, respectively elimination *vs* energy recovery, could be demonstrated in this doctoral thesis by technological processes used for several decades, combustion and pyrolysis, but brought in an updated and targeted form on the energy mixtures developed.

2. Original contributions on the production and energy recovery of solid sorts based on sewage sludge (SS), meat and bone meal (MBM), slag (Z) and biomass (BW; BS)

The present study aimed to overlap and correlate several topical issues: *(i)* sustainability of resources, *(ii)* environmental protection, *(iii)* elimination through recovery - complementary themes, with the stated aim of developing innovative solid energy sorts (ESS), assimilated to alternative fuels, starting from the combination of wastes such as sewage sludge (SS), meat and bone meal (MBM), slag (Z) and biomass (BW; BS).

Table 1 presents the results on the moisture content (W), ash (A), volatiles matter (V) and chemicals elements C, N, H, S / O for waste raw materials (SS; MBM; Z; BW; BS), chosen in this study.

Table 1. Physico-chemical and energy characterization of waste raw materials (SS; MBM; Z; BW; BS) compared to lignite (L)

Determinations	L	SS	MBM	Z	BW	BS
W_t^i *(%)*	37,21 ± 4,50	77,90 ± 9,42	18,52 ± 2,24	39,76 ± 4,81	1,48 ± 0,18	1,48 ± 0,18
A^i *(%)*	28,20 ± 0,85	44,51 ± 1,34	6,49 ± 0,19	80,77 ± 2,42	13,86 ± 0,56	2,13 ± 0,06
V^i *(%)*	21,06 ± 0,63	51,13 ± 1,02	81,43 ± 2,44	10,59 ± 0,32	82,48 ± 2,24	78,79 ± 2,36
C^a *(%)*	21,29 ± 0,61	26,44 ± 0,76	28,95 ± 0,83	15,56 ± 0,45	0,34 ± 0,01	49,68 ± 1,43
N^a *(%)*	1,53 ± 0,05	4,62 ± 0,15	11,09 ± 0,37	0,26 ± 0,01	5,50 ± 0,58	1,42 ± 0,05
H^a *(%)*	2,06 ± 0,05	4,04 ± 0,10	6,34 ± 0,16	0,52 ± 0,02	< 0,005	6,42 ± 0,16
S^a *(%)*	0,45 ± 0,03	0,65 ± 0,04	8,88 ± 0,50	0,34 ± 0,02	36,60 ± 0,78	< 0,005
O^a *(%)*	9,24 ± 0,18	19,52 ± 1,79	38,25 ± 1,10	2,55 ± 0,06	3,66 ± 0,06	36,60 ± 1,10
FC^a *(%)*	50,74 ± 1,52	4,36 ± 0,09	12,08 ± 0,36	8,64 ± 0,26	44,00 ± 5,41	19,08 ± 0,57
Q_s^a *(kcal / kg)*	2086 ± 25	2915 ± 35	3730 ± 45	971 ± 12	4044 ± 51	4813 ± 58
Q_i^a *(kcal / kg)*	1700 ± 20	2698 ± 32	3400 ± 41	800 ± 10	3691 ± 55	4591 ± 55
EF *(kg / GJ t / TJ)*	94,12	83,62	71,55	-	0,00	0,00

Note: *i - initial state; a - analysis state*

The highest ash content (A) in the slag (Z) was predictable, this being a residual product resulting from the combustion of lignite. Also, the high level of ash (A) is found in the sewage sludge (SS) samples. Lignite, waste biomass (BW) and beech sawdust (BS) show ash levels (A) within limits comparable to those in the literature in the case of such matrices.

In the case of volatile matter content (V), the results were spectacular due to the proven potential of alternative fuels. The samples of sewage sludge (SS) and meat and bone meal (MBM) recorded high levels of volatile matters, compared to those of lignite, a fossil fuel used regularly in the boilers of power plants in southwestern Romania. If in the case of beech sawdust (BS), known as an alternative fuel, with proven high energy potential, in the case of waste-type biomass, respectively apricot kernels (BW), but also grape residues (BW), the results of energy tests have generated higher values than those reported in the literature.

By developing a solar dryer, Figure 1, the doctoral study demonstrated the possibility of using sewage sludge (SS) by combustion in the form of pellets, for a high efficiency of a material considered by modern society as waste.

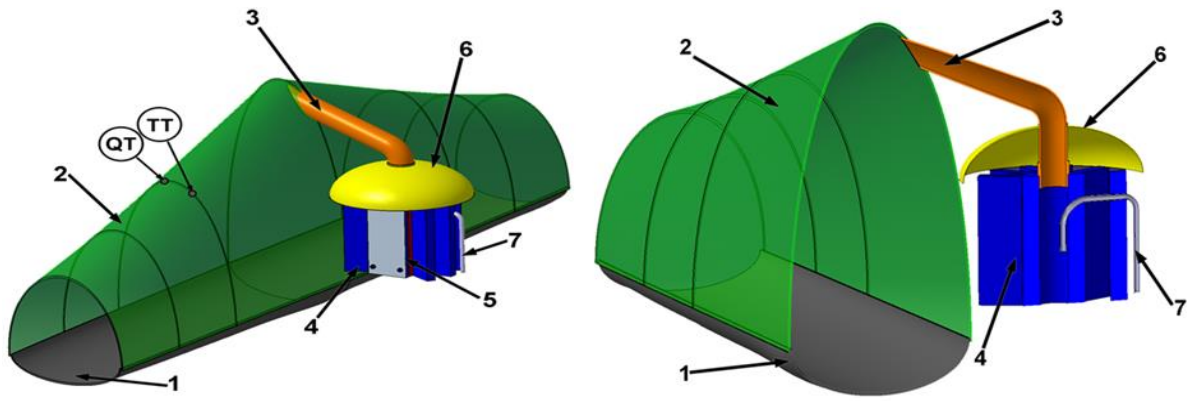

Figure 1. Solar dryer with moisture extractor, designed and used in experimental testing

The dryer, according to the invention, consists of a lower inner support element *(i)* for the components to be dried, of concave shape, which concentrates the sun's rays, and *(ii)* a solar concentrator, formed of an upper covering structure, dome-shaped, transparent, semicircular inclined, having a paraboloid shape with different diameters at the ends, compared to the middle section, together with which it forms a closed cavity, from which the resulting water vapor is extracted by static diffusion at the top with by means of a *(iii)* connecting tube, and are discharged by natural or forced condensation by means of a *(iv)* element with metal fins, and by the possibility of free or forced cooling with a *(v)* Peltier type element, equipped with a *(vi)* glossy, umbrella-shaped covers, the condensate being discharged from the condensing chamber through a *(vii)* tube operating on the principle of communicating vessels and hydrostatic pressure. The **QT** and **TT** elements are used for the measurement and quantitative verification of humidity, but also of the gas concentration resulting during the drying operation under the influence of sunlight, the component elements of the installation being arranged in a certain sequence and configuration, forming a closed enclosure allow the human operator to accelerate the extraction of moisture as well as to follow the operating parameters, including the content and composition of the gases released during the drying process.

Also, the study on slag (Z) resulting from the combustion of lignite and in the innovative tricomponent mixture developed with lignite, Table 2, but also with a special type of biomass, meat and bone meal (MBM), contributed to the validation of the process of elimination of some waste with negative effects on the environment, quality of life in general, through combustion processes, perfectly feasible in high-capacity boilers of Romanian CHPs, without the risk of developing toxic chemicals, such as dioxins and furans, respectively polycyclic aromatic hydrocarbons (PAH).

Table 2. Types of innovative solid energy sorts (ESS) developed

Mixture type	Mass composition (%)							Mixture code
	SS	L	BS	Z	MBM	BW	BS	
two components	30	70						ESS$_{SS+L\ (I)}$
	50	50						ESS$_{SS+L\ (II)}$
	70	30						ESS$_{SS+L\ (III)}$
	30		70					ESS$_{SS+BS\ (I)}$
	50		50					ESS$_{SS+BS\ (II)}$
	70		30					ESS$_{SS+BS\ (II)}$
three components	25	50		25				ESS$_{SS+L+Z\ (I)}$
	50	25		25				ESS$_{SS+L+Z\ (II)}$
	25	25		50				ESS$_{SS+L+Z\ (III)}$
		50		25	25			ESS$_{MBM+L+Z\ (I)}$
		25		25	50			ESS$_{MBM+L+Z\ (II)}$
		25		50	25			ESS$_{MBM+L+Z\ (III)}$
		25			25	50		ESS$_{MBM+BW+L\ (I)}$
		25			50	25		ESS$_{MBM+BW+L\ (II)}$
		50			25	25		ESS$_{MBM+BW+L\ (III)}$
		50		25			25	ESS$_{BS+L+Z\ (I)}$
		25		25			50	ESS$_{BS+L+Z\ (II)}$
		25		50			25	ESS$_{BS+L+Z\ (III)}$

2.1 Two-component innovative solid energy sorts (ESS)

Table 3 shows the average values of the elemental chemical composition of the two-component innovative solid energy sorts (ESS) developed.

Table 3. Characteristics of two-component innovative solid energy sorts (ESS)

Parameters investigated	Type of two-component ESS sorts					
	ESS$_{SS+L\ (I)}$	ESS$_{SS+L\ (II)}$	ESS$_{SS+L\ (III)}$	ESS$_{SS+BS\ (I)}$	ESS$_{SS+BS\ (II)}$	ESS$_{SS+BS\ (III)}$
C^a *(%)*	30,11 ± 0,86	28,85 ± 0,83	27,14 ± 0,78	40,12 ± 1,15	33,91 ± 0,97	30,76 ± 0,88
N^a *(%)*	1,82 ± 0,06	2,28 ± 0,08	3,07 ± 0,10	1,08 ± 0,04	2,59 ± 0,09	2,97 ± 0,01
H^a *(%)*	3,61 ± 0,09	3,72 ± 0,09	3,92 ± 0,10	5,14 ± 0,13	4,67 ± 0,12	4,43 ± 0,11
S^a *(%)*	1,09 ± 0,06	1,15 ± 0,06	1,02 ± 0,06	0,26 ± 0,01	0,50 ± 0,03	0,67 ± 0,04
O^a *(%)*	22,29 ± 0,44	23,24 ± 0,46	24,78 ± 0,50	39,17 ± 0,78	37,18 ± 0,74	32,34 ± 0,65
FC^a *(%)*	17,47	36,99	10,99	13,73	13,51	10,29
Q_s^a *(kcal / kg)*	3019 ± 36	2916 ± 35	2814 ± 0,33	3807 ± 46	3499 ± 42	3200 ± 38
Q_i^a *(kcal / kg)*	2566 ± 31	2479 ± 30	2392 ± 29	3510 ± 42	3223 ± 39	2982 ± 36
EF *(kg / GJ t / TJ)*	91,942	91,206	88,91	97,15	89,341	88,614

In the literature there are studies on the realization of energy mixtures based on sewage sludge (SS) + coal, sewage sludge (SS) + biomass (sawdust, aquatic plants, mushrooms), in compositional proportions of 0 %; 3 %; 5 %; 10 %; 20 %; 30 % for SS: coal, respectively 0 %; 25 %; 50 %; 75 %; 100 % for SS: biomass, which are different compared to those developed in this study, **I** - 30 % + 70 %; **II** - 50 % + 50 %; **III** - 70 % + 30%.

Following the investigations, the results of the sulfur content showed that both the waste raw materials (SS; BS) chosen for the study and the two-component innovative solid energy sorts (ESS) developed fall within value thresholds of ~ 1 %.

Regarding the nitrogen (N) content, it should be emphasized that the sewage sludge (SS) has been used in the past as compost and fertilizer, precisely because of the high content of this element, in addition to the high content of phosphorus (P) and potassium (K). The nitrogen content of ~ 4.62 % present in sewage sludge (SS) and ~ 1.53 % in lignite varied in the developed two-component innovative solid energy sorts (ESS), as follows: 1.08 % for $ESS_{SS + BS (I)}$ and 3.07 % respectively for $ESS_{SS + L (III)}$, which represents a progress in reducing its level, respectively of nitrogen oxides (NO_x) resulting from combustion. In the cases recorded by the literature in which a coal with a higher nitrogen level, N ~ 2 %, was used, it was also reflected in the final concentration of the developed energy mixtures.

The carbon content (C) can provide important information not only about the energy potential of the investigated materials, but also about the potential emissions of CO_2, GHG, responsible for the global greenhouse effect - Global warming, through the future combustion of innovative solid energy sorts (ESS). The energy potential of waste raw materials (SS; BS) selected in this study, can increase through the proposed energy sorts, without negatively affecting the environment through the level of emissions developed in combustion processes.

After removing the water from the samples by drying in special ovens followed by cutting, grinding and sieving operations at a specified specific granulation, their combustion potential was investigated by determining the higher calorific value (Qs) and lower calorific value (Qi) using the calorimetric method. The values obtained are high in terms of combustion, especially for the $ESS_{SS + BS (I; II; III)}$ sorts. Compared to the value of the superior calorific value of the sewage sludge (Qs = 2915 kcal / kg), respectively of the lignite (Qs = 2100 kcal / kg), the innovative solid energy sorts (ESS) demonstrated values of the superior calorific value between 2814 kcal / kg $ESS_{SS + L (III)}$ and 3807 kcal / kg $ESS_{SS + BS (I)}$, which is a feasible solution for the disposal of solid waste with energy recovery.

The literature specifies the following energy values for energy sorts developed from sewage sludge (SS) and biomass, respectively aquatic plants, *(i)* SS + water hyacinth, Qs ~ 2521-2785 kcal / kg and *(ii)* SS + sedge, Qs ~ 2774-3362 kcal / kg. Also, in the case of energy sorts based on sewage sludge (SS) and coal, the literature indicates Qs values, respectively 1931-3852 kcal / kg, using a coal type coal (Qs = 6128 kcal / kg). Although for the innovative solid energy sorts (ESS) based on lignite $ESS_{\mathbf{SS+L\ (I;\ II;\ III)}}$ developed in this study, the energy values are close to those in the study in which another type of coal, coal was used, the difference is in the ratio of waste components used in energy sorts, to the advantage of ESS.

2.2 Innovative three-component solid energy sorts (ESS)

In the literature no data were identified regarding the development of energy mixtures with three components of waste raw materials. Consequently, the design and development of three-component innovative solid energy sorts (ESS) in this doctoral study is a novelty.

Tables 4-5 present the average values of the elementary chemical composition of the innovative three-component solid energy sorts (ESS) developed and tested in the present doctoral study.

Table 4. Physico-chemical and energetic characteristics of the innovative solid energy sorts of three-component ESS

Parameters investigated	Type of three-component ESS sorts					
	$ESS_{\mathbf{MBM+L+Z\ (I)}}$	$ESS_{\mathbf{MBM+L+Z\ (II)}}$	$ESS_{\mathbf{MBM+L+Z\ (III)}}$	$ESS_{\mathbf{MBM+BW+L\ (I)}}$	$ESS_{\mathbf{MBM+BW+L\ (II)}}$	$ESS_{\mathbf{MBM+BW+L\ (III)}}$
C^a *(%)*	25,32 ± 0,40	26,91 ± 0,36	22,31 ± 0,34	32,66 ± 0,46	34,99 ± 0,46	34,22 ± 0,51
N^a *(%)*	3,22 ± 0,17	5,10 ± 0,55	2,94 ± 0,51	3,44 ± 0,69	6,20 ± 0,94	5,51 ± 0,02
H^a *(%)*	3,78 ± 0,42	4,40 ± 0,49	2,98 ± 0,44	4,77 ± 0,86	5,59 ± 0,51	5,61 ± 0,47
S^a *(%)*	2,20 ± 0,55	2,00 ± 0,46	2,00 ± 0,30	2,16 ± 0,34	3,80 ± 0,67	4,96 ± 0,76
O^a *(%)*	18,06 ± 0,73	14,84 ± 0,77	14,84 ± 0,64	28,47 ± 0,94	32,36 ± 1,00	34,05 ± 0,98
FC^a *(%)*	13,42 ± 0,11	12,14 ± 0,17	11,46 ± 0,19	15,28 ± 0,12	15,18 ± 0,21	17,08 ± 0,18
Q_s^a *(kcal/kg)*	2493 ± 30	2791 ± 33	2162 ± 26	3412 ± 41	3759 ± 45	3982 ± 48
Q_i^a *(kcal/kg)*	2120 ± 25	2433 ± 29	1876 ± 23	3129 ± 38	3444 ± 41	3600 ± 43
EF *(kg/GJ t/TJ)*	93,628	88,883	95,128	56,223	98,123	67,277

Note*: (a) - state of analysis*

Table 5. Physico-chemical and energetic characteristics of the innovative solid energy sorts of three-component ESS

Parameters investigated	Type of three-component ESS sorts					
	ESS_{BS+L+Z} (I)	ESS_{BS+L+Z} (II)	ESS_{BS+L+Z} (III)	ESS_{SS+L+Z} (I)	ESS_{SS+L+Z} (II)	ESS_{SS+L+Z} (III)
C^a *(%)*	27,31 ± 0,78	33,46 ± 0,96	24,12 ± 0,69	23,90 ± 0,69	24,06 ± 0,69	20,44 ± 0,59
N^a *(%)*	0,89 ± 0,03	0,77 ± 0,03	0,72 ± 0,02	1,68 ± 0,06	2,66 ± 0,09	1,67 ± 0,06
H^a *(%)*	3,30 ± 0,08	4,07 ± 0,10	2,65 ± 0,07	3,00 ± 0,08	3,50 ± 0,12	2,80 ± 0,07
S^a *(%)*	0,35 ± 0,02	0,18 ± 0,01	0,16 ± 0,01	0,79 ± 0,04	0,40 ± 0,02	0,24 ± 0,01
O^a *(%)*	21,73 ± 0,43	29,74 ± 0,59	18,55 ± 0,37	13,70 ± 0,27	14,73 ± 0,29	10,59 ± 0,21
FC^a *(%)*	13,73	13,51	10,29	10,83	8,87	10,39
Q_s^a *(kcal/kg)*	2673 ± 0,16	3219 ± 39	2403 ± 29	2299 ± 28	2410 ± 29	1936 ± 23
Q_i^a *(kcal/kg)*	2323 ± 28	2951 ± 35	2189 ± 26	1978 ± 24	2122 ± 25	1660 ± 20
EF *(kg/GJ t/TJ)*	94,486	95,823	92,531	95,835	92,033	97,328

***Note**: (a) - state of analysis*

The three-component innovative solid energy sorts (ESS) consisting of meat and bone meal (MBM), $ESS_{MBM + L + Z}$, respectively $ESS_{MBM + BW + L}$, are sorts with a high energy level, Qs = 2483-3982 kcal / kg. The high level of sulfur (S), is a characteristic of the sorts that have in their composition meat and bone meal (MBM), due to the sulfur aminoacids present. This phenomenon is also reported in other studies, and a solution to correct SO_2 emissions would be to include a sustainable adsorbent, such as limestone.

In the present study, slag (Z) was used in three-component sorts with lignite, meat and bone meal (MBM) or sewage sludge (SS), biomass waste (BS), in different mass proportions. Low levels of nitrogen (N) (0.72-3.44%) and sulfur (S) (0.16-2.16%) in slag (Z), correlated with the appreciable level of carbon (C) (~ 24 -33%), for a waste raw material resulting from a combustion, are factors of interest for the introduction into the energy circuit, in energy sorts with other waste raw materials. All innovative solid energy sorts (ESS) developed on the basis of slag (Z) revealed a high level of calorific value, some at the same level or even higher compared to that of lignite, respectively Qs = 1936-3412 kcal / kg *vs.* 2086 kcal / kg fossil fuel used in the boilers of thermal power plants in the South-West of Romania, respectively CET Govora, CET Turceni, CET Ișalnița, CET Rovinari.

Sewage sludge (SS) used in a single combination of tricomponent innovative solid energy sort $ESS_{SS + L + Z}$, together with lignite and slag (Z), at different mass concentrations **(I; II; III)**, offers a new perspective, of the method of disposal of this waste raw material by energy recovery, exclusively through the combustion process. With varying energy values, Qs of $ESS_{SS + L + Z}$ **(I; II; III)** with values between 1936 kcal / kg and 3412 kcal / kg, compared to those of lignite Qs of 2086 kcal / kg and sewage sludge (SS) Qs of 2915 kcal / kg, sorts based on sewage sludge (SS) are energetically solid sorts and due to the high content of volatile matters (V) $ESS_{SS + L + Z (I; II; III)}$: 25,35-56,22 %, but also environmentally friendly, with nitrogen levels (N) $ESS_{SS + L + Z (I; II; III)}$ of 1.67-3.44 % and sulfur (S) $ESS_{SS + L + Z (I; II; III)}$ of 0.24-2.16 %.

2.3 Practical and efficient way to capitalize on innovative solid energy sorts (ESS) by making pellets

For a better research of the behavior of the innovative solid energy sorts (ESS) developed in this study, the best simulation is the real representation in the form of a pellet or a lighter. In a first stage, the exclusive pelletization of the sewage sludge (SS) was carried out, dried, ground, homogenized and subjected to the sieving process up to Ø < 200 μm. In this study were also developed pellets from a mixture of sawdust and sewage sludge, $ESS_{SS + BS (II)}$ shown in Figure 2, respectively lignite and sewage sludge, $ESS_{SS + BS (II)}$.

Figure 2. Pellets from sewage sludge + beech sawdust $ESS_{SS + BS (II)}$

Processed waste in the form of pellets allows its storage without leading to secondary environmental pollution. In addition, this shape facilitates transport, increasing their potential for use.

By performing tests for the physical properties on the developed skins, presented in Table 6, it is considered that they can be subjected to the operations of mechanical handling, transport, loading, unloading, in safe conditions.

Table 6. Technical and physico-chemical characteristics of the pellets developed from the innovative solid energy sorts (ESS)

Properties	Peletts ESS_{SS}	Peletts $ESS_{SS + BS (II)}$	Peletts $ESS_{SS + L (II)}$
color	black	ash	gray
smell	specific	specific	specific
surface	smooth	smooth	smooth
additive / binder	missing / without	missing / without	missing / without
compressive strength *(kN)*	~ 4,596	~ 4,800	~ 5,500
moisture content W *(%)*	~ 15	~ 15	~ 15
ash content A (%)	~ 44	~ 21	~ 40
lower energy value Q_i *(kcal/kg)*	~ 2540	~ 3300	~ 2500
diameter D *(mm)*	~ 5	~ 5	~ 5
length L *(mm)*	> 30	> 30	> 30
sulfur S *(%)*	~ 0,5	~ 0,5	~ 1
nitrogen N *(%)*	~ 4	~ 2	~ 1
volatile matter content V *(%)*	~ 50	~ 65	~ 25
density ρ *(kg/m^3)*	~ 1,2	~ 1,3	~ 1,0

2.4 Validation of the energy potential of ESS pellets

Waste processed in the form of pellets allows its storage without leading to secondary environmental pollution. In addition, this shape facilitates transport, increasing their potential for use. To validate the energy potential of the pellets developed in this study, their combustion tests were performed, Figure 3.

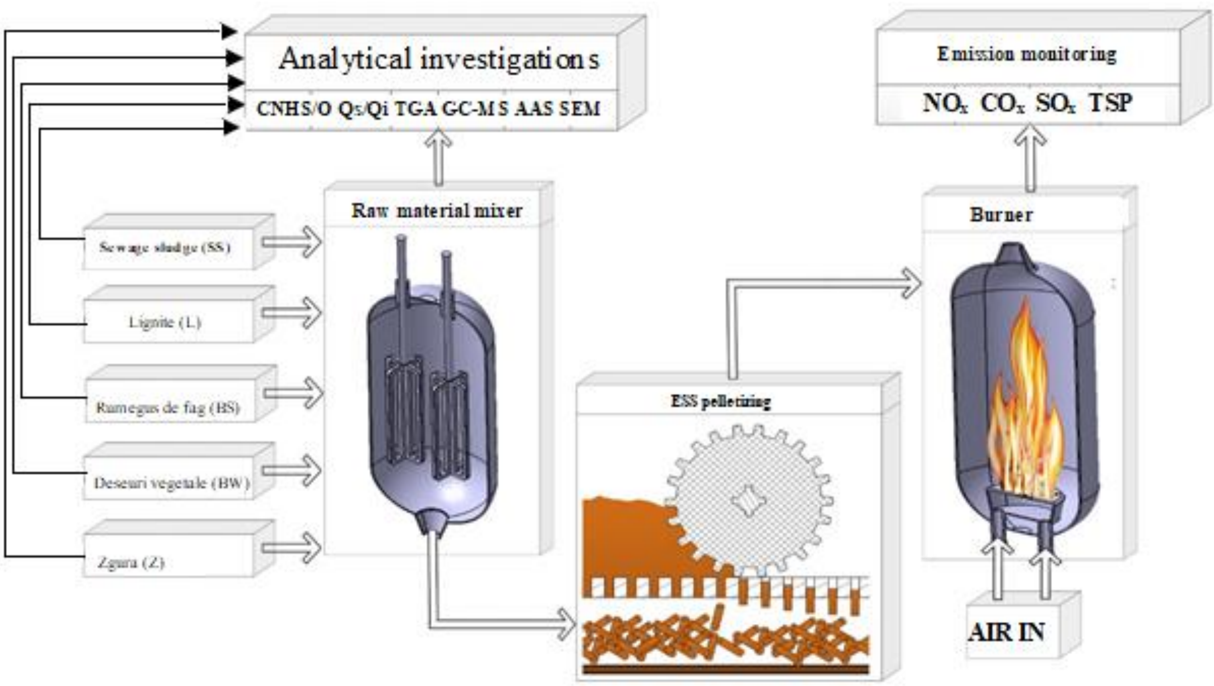

Figure 1. Flow diagram on waste recovery by pelletizing and combustion

Combustion of sewage sludge (SS), meat and bone meal (MBM), slag (Z) from the combustion of lignite, alone and / or in mixtures with lignite coal or beech sawdust biomass (BS), or waste biomass of plant origin (BW) - combines a number of advantages, which include a large reduction in the huge volume of ever-increasing waste.

The combustion characteristics of alternative fuels studied by thermogravimetric analysis and gas composition analysis by gas chromatography, respectively elemental analysis, but also by calorimetric investigations, showed a solid behavior, sustainable waste management through innovative solid energy sorts (ESS) developed. The combustion of innovative solid energy sorts (ESS) developed from the selected waste results in a good and promising alternative for the correct management of current resources, but also of waste, from disposal to energy recovery, respectively by:

(i) drying the selected waste in the solar dryer designed in this study, up to a humidity W < 15 %;

(ii) the higher calorific value (Qs) obtained, with values between 2500 kcal / kg and 4000 kcal / kg *vs.* 2200 kcal / kg resulting from lignite;

(iii) the values of gaseous emissions and the content of total dust generated by the combustion of pellets based on sewage sludge (SS), which are lower compared to the values of attention contained in Order no. 756 of November 26, 2004;

(iv) the total equivalent toxic concentration (TEC) of 0,48765 ng / kg, which is lower than that set for the cleaning level of 13 ng / kg;

(v) the sum of the metal content of the gaseous emissions resulting from the combustion of ESS, of 244.23 μg / kg, being lower than the one provided by Order no. 756/2004, of 500 μg / kg;

(vi) the potential use of combustion ash (SS_{ASH}) in construction materials.

3. Experimental research on the pyrolysis transformation of plastic waste

This doctoral study investigated from a thermochemical point of view the potential of several types of polymeric materials, polyethylene PE (HDPE and LDPE), polypropylene PP and polystyrene PS. The choice of these polymeric materials was based on the fact that they are "responsible" for > 90 % of the total plastic products worldwide, respectively for plastic waste.

In this study we proposed pyrolysis as a waste disposal process.

Pyrolysis is a process of chemical decomposition that takes place in an atmosphere without oxygen. Pyrolysis involves the fragmentation of polymers in an inert medium using reducing and oxidizing agents, in the presence or absence of a catalyst, at relatively high temperatures and atmospheric pressure. During this thermochemical process, chemical reactions of *(i)* hydrogenation, *(ii)* dehydrogenation, *(iii)* cracking, *(iv)* aromatization, *(v)* cyclization take place, finally new molecules developing. The complex pyrolysis process leads to the thermal decomposition of solid polymeric matrices into reaction products in three aggregation states: *(i)* solid - residue / wax; *(ii)* liquid - rich in hydrocarbons / condensed gases; *(iii)* gas - rich in hydrocarbons and other non - condensable gases.

3.1 Experimental pyrolysis plant

The pyrolysis plant has the following technological structure: *(i)* reactor - made of refractory steel, with H = 500 mm and Dint = 100 mm; *(ii)* reactor insulation made with basalt wool in a layer of l = 50 mm; *(iii)* metal condenser made of copper - cooling system / uncondensed gases with L = 500 mm; *(iv)* metal condenser cooling provided with water at T < 10 ° C; *(v)* glass - Erlenmeyer flask / condensable hydrocarbon collection / PPO type oil; *(vi)* bag of special material that does not allow the diffusion of non-condensable gases, V = 5 L Tedlar (CEL Scientific Corporation) for the collection of non-condensable gases, PPG type gas.

The development and validation through this study of alternative fuels such as pyrolysis oils - PPO, respectively pyrolysis gas - PPG, created the premises for sustainable waste management. The high energy value of the reaction products resulting from the studied pyrolysis processes was facilitated by the innovative structure of a developed reactor, which was the subject of a national patent granted by OSIM, Figure 4, used at lower temperatures compared to those presented in specialty literature.

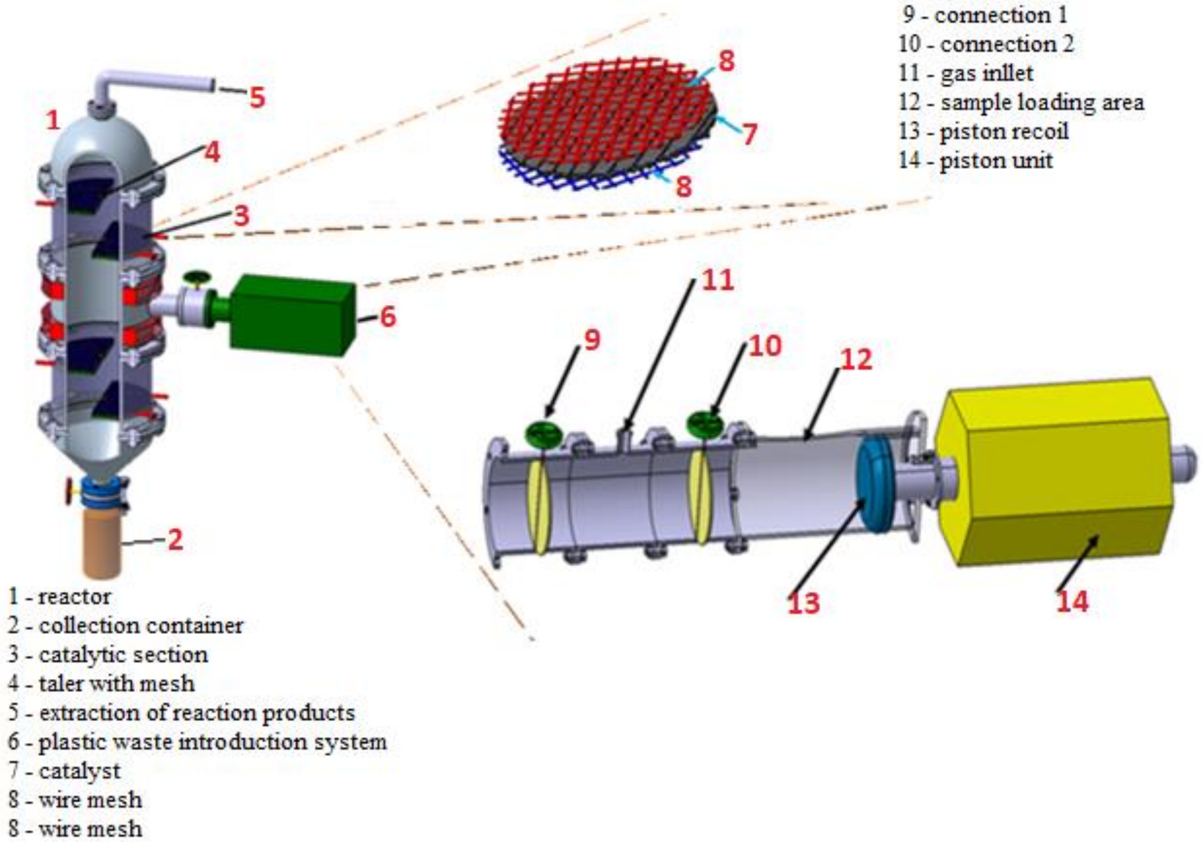

Figure 4. Reactor diagram of the experimental pyrolysis plant developed in this study

3.2 Physico-chemical and energetic characterization of pyrolysis oils (PPO) developed from plastics

Higher calorific value (Qs) determined following catalytic or non-catalytic pyrolysis processes, in the case of PP indicates values between 40.59 MJ/kg ($PPO_{PP + MCM41\ 450}$) and 44.95 MJ/kg ($PPO_{PP\ 350}$), the results being superior or similar to those in the literature for plastic waste made of the same type of polymer, PP, respectively: 40.80 MJ/kg, 44.37 MJ/kg, or 41.15 MJ/kg.

For the pyrolytic oils obtained as a result of the complex pyrolysis processes of PS, high energy values were recorded, slightly below those in the literature, Qs = 43 MJ/kg and 43.94 MJ/kg.

According to data from the literature, the pyrolysis process at 500 ° C performed with a reactor similar to that used in the present study, but in which the catalyst method was used mixed with plastic waste, in a mass ratio of 1:3, led to the following results: the value of the higher calorific value Qs = 42.12 MJ/kg for the pyrolytic oil of LDPE and of 43.19 MJ/kg for the pyrolytic oil of HDPE. Comparatively, in this doctoral study, at the catalytic mass: plastic ratio of 1:10, at a temperature of 450 °C, the values of higher calorific value varied from Qs =

42.94 MJ/kg in the catalytic process with MCM- 41 at Qs = 44.30 MJ/kg in the catalytic process with Zeolite. In another study, the HDPE polymer subjected to a process of non-catalytic pyrolysis at T > 475 °C led to a higher calorific value Qs = 41.15 MJ/kg, compared to the pyrolytic oil $PPO_{\mathbf{HDPE\ 450}}$, obtained in this doctoral study by non-catalytic pyrolysis with a much higher calorific value, of Qs = 44.50 MJ/kg.

The physical properties of the pyrolytic oils (PPO) obtained are shown in Table 7.

Table 7. Physical characteristics of oils resulting from the pyrolysis of plastics at 450 ºC

Type of pyrolytic oil	Observations and physical characteristics
$PPO_{HDPE\ 450}$	color - yellow, greenish appearance - oily, viscous inflammable odor - strong hydrocarbons / specific for conventional liquid fuels density at 25 °C = 7595 g/ cm^3
$PPO_{LDPE\ 450}$	color - yellow, greenish appearance - oily, viscous inflammable odor - strong hydrocarbons / specific for conventional liquid fuels density at 25 °C = 7478 g/ cm^3
$PPO_{PP\ 450}$	color - yellow aspect - oily inflammable odor - strong hydrocarbons / specific for conventional liquid fuels density at 25 °C = 7606 g/ cm^3
$PPO_{PS\ 450}$	color - red, brown appearance - viscous / semi-solid inflammable odor - strong hydrocarbons / specific for classic liquid fuels + styrene density at 25 °C = 9139 / cm^3 ETBE - 2,19 %

The S content of all samples is below the limit of quantification of the investigation method, respectively LOQ = 0.005 %. In this sense, the possibility of forming compounds with S content, respectively H_2S, SO_x, is very low, which makes the pyrolytic oil (PPO) to be environmentally friendly.

Also, the nitrogen (N) content is very low, being slightly influenced by the presence of the catalyst. On the other hand, the highest carbon content was 88.13 % recorded for PPO obtained from PS ($PPO_{\mathbf{PS\ MCM\text{-}41}}$), influenced by the MCM-41 catalyst. Also, the highest Qs was determined for PPO based on LDPE polymer ($PPO_{\mathbf{LDPE\ +\ Zeolite\ 450}}$), being 45.36 MJ/kg.

3.3 Heavy metal content of pyrolysis oils

Determining the heavy metal content of pyrolytic oils developed in this study of pyrolysis of plastics based on PP, HDPE, LDPE and PS provides important information to assess the impact on the environment, but also to compare them with levels of conventional liquid fossil fuels. The results of the heavy metal content of PPO developed in relation to the levels of conventional liquid fuels are presented in Table 8.

Table 8. PPO heavy metal content of the polymers investigated compared to that of conventional fuels

Fuel type	Cd (µg/ l)	Pb (µg/ l)	Cr (µg/ l)	Mn (µg/ l)	Co (µg/ l)	Ni (µg/ l)	Cu (µg/ l)	As (µg/ l)	Se (µg/ l)	Hg (µg/ l)	Rb (µg/ l)	Sr (µg/ l)
PPO_{PP450}	0,12	0,09	1,97	0,29	0,21	0,56	0,06	0,37	1,61	< 0,01	0,17	0,06
$PPO_{HDPE\ 450}$	0,34	0,53	2,49	8,11	1,39	3,83	2,31	0,17	1,72	< 0,01	0,01	0,72
$PPO_{LDPE\ 450}$	0,05	0,06	3,11	0,01	0,07	4,56	1,75	0,35	2,40	< 0,01	0,21	0,05
PPO_{PS450}	0,07	0,07	10,03	0,37	0,16	0,02	0,27	0,30	2,12	< 0,01	0,22	0,15
Diesel fuel	15,00	10,10	8,60	-	-	-	17,70	-	-	-	-	-
Kerosene	13,30	4,10	3,30	-	-	-	19,80	-	-	-	-	-
Benzine	16,80	2,40	5,40	-	-	-	17,40	-	-	0,77	-	-

3.4 Physico-chemical and energetic characteristics of PPG developed from investigated plastics

The composition of PPG gas resulting from HDPE is influenced by the use of SMMS / catalysts. HDPE - mixture of polyethylene and polypropylene, but also of other hydrocarbons, during the pyrolysis process makes the transition from solid to gaseous state with a rich content in hydrocarbons, but also in hydrogen H_2, which gives the resulting gas an important energy intake, especially in catalytic pyrolysis processes, respectively with SBA-15 or MCM-41, H_2 being 5.16-5.70 %, compared to the initial concentration of ~ 2 %. According to the obtained results, the PPG gases obtained as a result of the pyrolysis processes of the HDPE polymer, in different technical conditions, the temperature of 350 °C, but also the absence of catalysts, determine a higher calorific value of Qs = 102.75 MJ/m^3, followed by $PPG_{\mathbf{HDPE}}$ at T = 450 °C, in the absence of catalysts, with Qs = 106.46 MJ/m^3. Although there are obvious changes in the PPG compositional structure following the catalytic pyrolysis processes, a higher energy value is observed in the case of LDPE-based PPG pyrolytic gases obtained in the absence of catalysts, for $PPG_{\mathbf{LDPE\ 450}}$, the Qs value being 121.18 MJ/m^3, and for $PPG_{\mathbf{LDPE\ 350}}$ Qs of 109.97 MJ/m^3. Polypropylene subjected to the pyrolysis process at 450 °C, without catalysts, showed at the end a $PPG_{\mathbf{PP\ 450}}$ - gas mixture rich in hydrocarbons, with a content > 43 % propylene, other alkanes and alkenes, but also free H_2. The results show that $PPG_{\mathbf{PP}}$

have a varied content in CO, CO_2, because even if the pyrolysis reaction takes place in the absence of oxygen, the carbon in the polymer structure reacts with intramolecular oxygen, possibly by the addition of dyes or other curing agents or elasticity. Methane undergoes major changes in concentration, from ~ 6.6 % in the case of $PPG_{\mathbf{PP\ 450}}$ to ~ 18 % under the catalytic action of Lignite (lignin) and MCM-41, respectively to ~ 13 % in the presence of SBA-15. The variable content of PP concentration, which decreases from ~ 43 % to ~ 36-39 %, also led to a decrease in energy values from 99.45 MJ/m^3 for $PPG_{\mathbf{PP\ 450}}$ to 80.86 MJ/m^3 for $PPG_{\mathbf{PP\ SBA15}}$, respectively 84.84 MJ/m^3 in the case of $PPG_{\mathbf{PP\ MCM41}}$. A generally valid feature for pyrolysis processes is that the future composition of the resulting gas depends in proportion > 80 % on the initial structure of the solid / liquid material subjected to the thermal degradation activity. Thus, the composition of PPG gas depends on the composition of the tested polymer, styrene and toluene being the main components identified by GC analysis in $PPG_{\mathbf{PS}}$ type gases. From the obtained data it can be seen how under the action of the cracking reaction during the pyrolysis process, but also of the catalysts, Zeolite, Lignite, SBA-15 and MCM-41, for toluene, the basic element of polystyrene PS, the benzene cycle undergoes a process of “breaking”, and a large part of the C and H content migrates to ethylene, and to a small extent to free H_2. This technical example is confirmed by changing the concentration level, initially from H_2 = 0.57 % in the case of $PPG_{\mathbf{PS\ 450}}$, to 2 % in the case of $PPG_{\mathbf{PS\ 450\ ZEOLITE}}$, $PPG_{\mathbf{PS\ 450\ LIGNITE}}$, $PPG_{\mathbf{PS\ 450\ SBA15}}$. An enrichment of the styrene concentration level was found in all cases of PPG obtained under catalytic action. For PPGs obtained from catalytic and non-catalytic pyrolysis experiments, a number of physical properties similar to those of classical gaseous hydrocarbons were identified. The results are presented in Table 9.

Table 1. Physical properties of PPG resulting from the pyrolysis of plastics

PPG type	Physical observations
$PPG_{HDPE\ 450}$	color - whitish appearance - viscous very flammable odor - hydrocarbons / specific for classic liquid fuels
$PPG_{LDPE\ 450}$	color - whitish appearance - viscous very flammable odor - hydrocarbons / specific for classic liquid fuels
$PPG_{PP\ 450}$	color - colorless appearance - viscous very flammable odor - strong hydrocarbons, specific for conventional liquid fuels
$PPG_{PS\ 450}$	color - mixture of white and gray appearance - viscous very flammable odor - a mixture of styrene and gasoline

The results obtained on the developed alternative fuels, PPO and PPG, following complex investigations using modern methods such as EA, calorimetry, ATR-FTIR, GC-FID, GC-TCD, GC-MS, AAS and TGA, can be summarized as follows:

(i) the developed pyrolytic oils (PPOs) showed a high and varied level of chemicals considered combustible, respectively for H, O and C;

(ii) the developed pyrolytic oils (PPO) confirmed a high energy potential, with values Qs ~ 39.5-45.5 MJ / m^3, comparable to those of conventional or alternative liquid fuels, used on the world profile market;

(iii) the developed pyrolytic gases (PPG) are characterized by the lack of sulfur compounds, specific to natural gas or biogas, respectively H_2S, SO_2, which is another important quality, namely that in relation to environmental protection;

(iv) the developed pyrolytic gases (PPG) are characterized by a lack of nitrogen compound content, which limits NO_x and zero oxide emissions, respectively;

(v) the developed pyrolytic gases (PPG) have proven energy values comparable to any gaseous fuel, both conventional and alternative, with Qs values of 80-110 MJ/m^3 *vs.* 42 MJ/m^3 for natural gas (NG);

(vi) the use of catalysts in sandwich steel blocks, SMMS, in catalytic pyrolytic processes, has brought economic efficiency, their recovery for other such experiments, with a yield η ~50 %;

(vii) the use of the SMMS catalytic system did not result in higher energy values of PPG compared to the non-catalytic process, instead it led to a significant reduction in the amounts of greenhouse gases, respectively CO_2 and CO, at a level < 100 ppm;

(viii) by burning alternative fuels developed in this study, respectively PPO and PPG, no chlorine emissions will be generated, because the polymers investigated, PP, PS, HDPE and LDPE do not contain chlorine following the EA type investigations performed;

(ix) carcinogenic heavy metal content determined from PPO *vs*. liquid classic fuels, is at least an order of magnitude smaller; thus, for Pb the content was between 0.07 μg/l and 0.53 μg/l ($PPO_{\mathbf{HDPE}}$) *vs.* 2.4-10.10 μg/l (diesel);

(x) the presence of tert-butyl ether (ETBE) has been identified in the developed $PPO_{\mathbf{PS\ 450}}$;

(xi) the level of PPW waste resulting from the pyrolysis of the polymers studied is relatively low < 10 % compared to the total amount of sample entering the experiment;

(xii) as regards the physical characteristics of PPO, the developed pyrolytic oils on the basis of HDPE, LDPE and PP, showed a yellow, yellow-green color, with a strong hydrocarbon odor and an oily / viscous appearance; $PPO_{\mathbf{PS\ 450}}$ oils are reddish in color, very viscous / semi-solid; all developed oils are highly volatile;

(xiii) the developed PPGs have a viscous / milky, hydrocarbon-smelling and highly volatile appearance;

(xiv) the developed pyrolytic oils (PPO) can find applications in high-capacity power and thermal power plants, as well as in wall-mounted ones, with or without refining or distillation processes; also, the developed pyrolytic gases (PPG) in this study can be used in thermal and power plants;

(xv) FTIR analysis allowed the identification of adsorption bands characteristic of pyrolytic oils $PPO_{\mathbf{HDPE450}}$ and $PPO_{\mathbf{LDPE450}}$, in the range of 2000 - 1700 cm^{-1}, 1600 - 1460 cm^{-1}, 1275 -1000 cm^{-1} and 900 - 690 cm^{-1}, fact which confirms the presence of aromatic groups in the compounds resulting from the pyrolysis processes.

Original contributions

The present study, based on the methodology used, the tests performed, and the encouraging results obtained is based on personal contributions, both theoretical and practical, materialized through publications such as articles, participation in international conferences, scientific projects won and proposals for applications. OSIM patent in the field, offering a "friendly" perspective on some raw materials known to today's society as waste, sewage sludge (SS), meat and bone meal (MBM), slag (Z), biomass (BS BW), polystyrene (PS), polypropylene (PP) and polyethylene (LDPE and HDPE).

Application contributions:

- development of an efficient technological system for solar drying of sewage sludge (SS), from W ~ 80 % to W < 15 %;
- the development of a technological system for the disposal of plastic waste through a catalytic and non-catalytic pyrolysis process, by means of a reactor equipped with sandwich blocks to support the catalysts;
- development of alternative fuels from plastics: *(i)* pyrolysis oil - PPO and *(ii)* pyrolysis gas - PPG;
- development of alternative fuels such as innovative solid energy sorts - ESS from waste raw materials;
- development of a pellet product from innovative solid energy sorts - EES developed.

The developed alternative fuels, ESS, PPO and PPG, can be a feasible alternative in high capacity thermal and / or electrical installations.

The success of such research in the field of alternative fuels obtained from waste raw materials is of interest to the economic environment, to private companies, investors and state-owned enterprises, with immediate visible effects on environmental protection, quality of life in general. The technological transfer to the economic environment is sustainable and could lead to social inclusion, the creation of new jobs.

Keywords: *waste, raw materials, capitalization, alternative fuels, energy, environment, sustainability.*

List of publications resulting from doctoral research, published or accepted for publication

Articles published in ISI listed journals

1. ***Marius Constantinescu,*** Simona Oancea, Felicia Bucura, Corina Ciucure, and Roxana Elena Ionete. Evaluation of the fuel potential of sewage sludge mixtures with beech sawdust and lignite. Journal of Renewable and Sustainable Energy. Vol. 10, 053106 doi.org/10.1063/1.5039808;(2018).

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Impact factor: 1,511

Citations: 2

2. ***Marius Constantinescu,*** Felicia Bucura, Roxana-Elena Ionete, Violeta-Carolina Niculescu, Eusebiu Ilarian Ionete, Anca Zaharioiu, Simona Oancea, Marius Gheorghe Miricioiu. Comparative Study on Plastic Materials as a New Source of Energy. Materiale plastice. Vol. 56, No. 1, pp. 41-46; (2019).

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Impact factor: 1,248

Citations: 2

3. ***Marius Constantinescu,*** Felicia Bucura, Eusebiu Ilarian Ionete, Daniela Ion-Ebrasu, Claudia Sandru, Anca Zaharioiu, Florian Marin, Marius Gheorghe Miricioiu, Violeta Carolina Niculescu, Simona Oancea, Roxana Elena Ionete. From Plastic to Fuel - New Challenges. Materiale plastice. Vol. 56, No. 4, pp. 721-729; (2019). https://www.revmaterialeplastice.ro/Articles.asp?ID=5259

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Articles published in ISI Proceedings

1. ***M. Constantinescu,*** F. Bucura, R. Ionete, A. Zaharioiu, C. Ciucure, S. Oancea. Sewage sludge eliminating by capitalizing on its energy potential. SGEM Conference Proceedings, ISBN 978-619-7408-28-7 / ISSN 1314-2704. Vol. 17, Issue 43, pp. 767774; (2017). https://www.sgem.org/sgemlib/spip.php?article11197

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1. ***M. Constantinescu,*** F. Bucura, R. Ionete, A. Zaharioiu, C. Ciucure, S. Oancea. SGEM Sewage sludge eliminating by capitalizing on its energy potential.

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OSIM patent granted

1. Ionete Roxana Elena, Ionete Eusebiu Ilarian, Spiridon Ştefan Ionut, ***Constantinescu Marius,*** Felicia Bucura, Marius Gheorghe Miricioiu. Plastic waste recovery plant. RO133259 (A0) - 2019-04-30. (2018)

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1. Ionete Roxana, Ionete Eusebiu Ilarian, Spiridon Stefan Ionut, ***Marius Constantinescu.*** Solar dryer with moisture extractor. No. of application: a 2018 00390. (2018)

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1. Plastic waste energy vector - ENERGPLAS. PN-III-CIRCLE-CO-CI-2018. Nr. 159 CI. Innovation CEC / UEFISCDI. Technical manager: ***Marius Constantinescu***. https://www.icsi.ro/energplas, (2018)

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1. Diploma of Excellence - International Exhibition of Scientific Research, Innovation and Inventions Pro Invent Edition XVII, March 20 - 22, 2019, Cluj Napoca, https://crmse.utcluj.ro/index.php/anunturi/salonul-pro-invent-2019-in-20-22-martie-lacluj-napoca.html

patent proposal "Solar dryer with humidity extractor", authors: Roxana Elenna Ionete, Ionete Eusebiu Ilarian, Stefan Ionuț Spiridon, ***Marius Constantinescu.***

2. Diploma of Excellence and INVENTICA Medal 2019 - International Invention Exhibition “INVENTICA 2019”, 26-28.06.2019, Iaşi, Romania

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