



## WASTE THERMOPLASTIC CHARACTERIZATION: GASIFICATION FOR USE IN MCI

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**Abstract.** *The clear control of the petroleum industry and their derivatives, associated with their huge variety of products produced from various types of plastics, and after consumed by ourself has generated a large amount of waste causing environmental and socio-economic problems due to their incorrect disposal. In this context, this study aims to the thermochemical characterization of the thermoplastics waste like the polyethylene, considering the utilization as a material for co-gasification. Elemental analysis shows only amounts of carbon, hydrogen and oxygen. The proximate analysis, due to the lack of standards for this kind of fuels, it was possible only determines quantities of moisture and ash, of about 0,3% and 0,5% respectively. The gross calorific value of the samples tested was between 40 - 45 MJ kg<sup>-1</sup>. Assays of Thermogravimetry and Differential Scanning Calorimetry were also performed. The ramp of temperature was 30-700 °C with three heating rates (2 Kmin<sup>-1</sup>, 20 Kmin<sup>-1</sup>, 40 Kmin<sup>-1</sup>, 100 Kmin<sup>-1</sup>) at two atmospheres, inert (N<sub>2</sub>) and oxidant (air). The results show agreement with the proximate analysis for moisture and ash. The contents of fixed carbon and volatile materials were determined by mass balance, comparing the results of the analyzes. Due to the large amount of volatile material found, the use of this type of waste suggests co-gasification with carbonaceous material to achieve pyrolysis temperatures and ensuring a self-sustaining process.*

**Keywords:** Waste, Plastic, Gasification, Thermal Analyzes, Fuel

### Nomenclature

BPE	Biodegradable Polyethylene	MSW	Municipal Solid Waste
DSC	Differential Scanning Calorimetry	PE	Polyethylene
HDPE	High Density Polyethylene	TG	Thermogravimetric Analysis
HHV	High Heating Value	VPE	Virgin Polyethylene
LHV	Low Heating Value	WRPE	Waste Recycled Polyethylene
LDPE	Low Density Polyethylene	WVPE	Waste Virgin Polyethylene

## 1. INTRODUCTION

The large amount of plastic artifacts produced and consumed in Brazil has contributed to the increasing production of Municipal Solid Waste (MSW), since a high percentage of these residues (13.5%) is attributed to the plastic (ABRELPE, 2012). In Brazil, the highest percentages of use of plastic materials are located in the areas of food packaging, construction, household appliances and others. In 2011, the apparent consumption of processed plastics was close to 6.894 million tons, where the polyethylene is the largest volume thermoplastic polymers in use today, around 43% (ABIPLAST, 2011).

Only 21.7 wt.% of the plastic waste produced is mechanically recycled, most of the remainder is disposed of in dumps or landfills. All of this material could be ignited in a controlled way and thus reduce by 90% the mass of the volume grounded, using its energy content and for generating alternative energy fuel gas (PLASTIVIDA, 2012).

The word recycling gained prominence in 1980s, when the first evidences of that the oil and other non-renewable materials exhaustion were noted, and the lack of space for waste disposal were finites. Plastics Europe (2012), highlights the growth rate of recycling waste plastics in European countries when compared to disposal of in landfills. This reuse that occurs in many different ways, symbolizes a global trend towards greater concern with the disposal of MSW generated, resulting in further studies and consequently the emergence of new technologies that provide the development of new techniques and procedures.

The Thermochemical Recycling is an important method of utilization of waste plastics, its goal is the recovery of chemical components that serve as fuel for energy and as a feedstock in refineries or petrochemical plants, in order to obtain high quality products (Carneiro, 2009).

The recovery of the energy contained in plastics by thermal processes have been investigated in several parts of the world, according to Carneiro (2009), the energy contained in 1 kg of plastic is equivalent to 1 kg of fuel oil. Besides saving and energy recovery, recycling occurs reducing in 70 to 90% the mass of the materials, leaving only an sterilized inert residue.

The main motivation of the thermal conversion is harnessing the thermal energy contained in the waste plastics. These, when mixed with other waste materials contribute to increase the calorific value, allowing a more complete combustion and higher temperatures (Al-Salem, et al., 2009).

Independent of the thermal conversion process that can be employed during the treatment of any waste material, particularly plastic materials, prior knowledge of their elemental composition, proximate and some other basic thermochemical behavior, i.e. thermogravimetry analysis (TG), it is fundamental understanding the mechanisms involved in each step of the process studied. Thus, it is possible to analyze and interpret the results, and formulate strategies to address the problem of safely and effectively.

Table 1 summarizes the results of proximate, calorific value and ultimate analysis found by several authors. Cozzani, et al., (1995) in his study on pyrolysis of MSW through a thermogravimetric and calorimetric approach, have characterized and developed a kinetic modeling for different materials considered key components (plastics, wood, paper). According to the authors, although some materials are fairly similar in their physical characteristics, the results may differ significantly, since the products obtained also depend on the route of production that is used. Also note that the presence of the plastic waste in the constitution strongly influences the combustion characteristics by changing the heat of reaction, the ignition properties and the final products obtained. They also report the need to establish new methods for the characterization of heterogeneous materials.

**Table 1.** Polyethylene Ultimate and Proximate analysis and Calorific Value

<b>Component</b>	Al-Salem, <i>et al.</i> apud Goto <i>et al.</i> , (2010)	Miranda, (2009)	Cozzani <i>et</i> <i>al.</i> , (1995)	Grammelis, <i>et al.</i> , (2009)	He <i>et al.</i> , (2009)	Kalanatarifard & Su Yang, (2012)	Park, <i>et al.</i> , (2012)
<b>C</b>	84.83	84.8	84.3	84.1	85.81	-	85.7
<b>H</b>	14.08	14.5	15.4	12.15	13.86	-	14.2
<b>N</b>	-	0.3	-	0.0	0.12	-	0.05
<b>O</b>	-	0.3	-	0.0	0	-	0,05
<b>S</b>	-	-	-	-	0.06	-	0
<b>Moisture</b>	-	0.0	-	0.1	0.02	0.54-0.82	0.3
<b>Volatile</b>	99.87	99.8	99.7	97.6	99.85	97.0	99.7
<b>Material</b>							
<b>Fixed Carbon</b>	-	0.1	0.1	2.3	0	12.0	0
<b>Ash</b>	0.13	0.1	0.2	0.0	0.15	1.4	0
<b>LHV</b> ( $MJkg^{-1}$ )	41.85	43.29	-	45.7	38.04	-	46.6

Thermogravimetric analysis in the field of polymeric materials is another technique that has been widely used since the 60's. Through this the reactions of dehydration, combustion, decomposition, among others, can be established. Sinfronio (2006) in order to check the PE thermal behavior carried out thermogravimetric tests in the temperature range of 30-900 °C at different heating rates (5, 10 and 20 °C  $min^{-1}$ ) under a nitrogen atmosphere ( $N_2$ ). The process of degradation has occurred between the temperatures 387-519 and 398-518 °C for LDPE and HDPE, respectively, and the maximum conversion achieved at temperatures of 490 and 492 °C. The curves exhibited only one decomposition step, wherein all of the polymer has been converted. Thermogravimetric analysis performed by Valle & Guimarães (2004) also showed that the thermal degradation occurs in a single stage, however, the process occurred between the temperatures 453-492 and 460-493 °C for LDPE and HDPE, respectively; the maximum conversion achieved at temperatures of 480 and 482 °C.

Candian (2007) in his study of high density polyethylene recycled for use in structural elements of PE samples characterized by thermal analysis and mechanical. The amount of ash at 600 °C was 1.77 %. According to the author, this small percentage of ash may correspond to the inorganic pigment or a lubricant additive employed during the manufacture of plastic. The authors also performed Differential Scanning Calorimetry experiments (DSC), and observed only one exothermic peak around 135.49 °C. This peak was attributed to the melting point of the material.

Coelho *et al.*, (2010) TG/DSC experiments PE samples, either pure or mixed with the catalysts. The DSC signal showed two endothermic peaks; the first one is around 136 °C and 119 °C corresponds to the melting temperature of the HDPE and LDPE samples, respectively. As expected, these peaks are not accompanied by any weight change. A second endothermic peak occurs at higher temperatures and corresponds to the degradation of the polymers and thus, it is accompanied by a significant weight loss.

## 2. POLYETHYLENE THERMOCHEMICAL CHARACTERIZATION

A series of analyzes were performed in samples of virgin polyethylene (VPE), waste recycled polyethylene (WRPE), waste virgin polyethylene (WVPE) and biodegradable polyethylene (BPE), where the main objective was to investigate such materials, in order to describe and identify its thermal characteristics and chemical composition. The VPE and BPE comes from petrochemical industry. The WRPE and WVPE samples were obtained from municipal solid waste in the city of Vitoria - ES.

**Table 2.** Experimental results for Polyethylene Ultimate and Proximate analysis (mean values in wt%)

Component	VPE	WVPE	WRPE	BPE 1	BPE 2
<b>C</b>	85,7	85,4	84,3	57,2	63,6
<b>H</b>	14,3	14,3	13,7	9,8	9,6
<b>N</b>	0	0	0	0	0
<b>O</b>	0	0,3	2,0	33,0	-
<b>S</b>	0	0	0	0	0
<b>Moisture</b>	0,05	0,17	0,20	1,57	0,24
<b>Ash</b>	0,25	0,37	1,05	0,08	16,52
<b>LHV (MJKg<sup>-1</sup>)</b>	46,2	46,0	44,6	25,2	-

The results of elemental and proximate analyses, as well as the calorific value are listed in Tab. 2. The VPE and WVPE elemental analysis indicates the presence mainly of carbon and hydrogen. The oxygen obtained by the difference can be attributed to possible contaminants present in the samples recycled, for example, pigments, plasticizers and other types of plastics. Note that the uncertainty quantification of oxygen becomes higher, since it was considered that there is no other chemical elements. The BPE samples have a smaller C/H ratio, and a higher amount of other elements.

The proximate analysis indicates just moisture and ash component. The samples with higher ash content were the WRPE and BPE2. While the moisture content generally is inconsiderable.

The high heating value (HHV) and low heating value (LHV) were determined according to the method Dulong (Perry and Chilton, 1973), Equation (1) and (2).

$$LHV = HHV - 600(9H + W) \quad (1)$$

Where,

$$HHV = 8100 C + 34000 \left( H - \frac{O}{8} \right) + 2500 S \quad (2)$$

In these equations, C, H, O, and S are the mass fractions of carbon, hydrogen, oxygen, and sulfur, obtained from elemental analysis, and W represents the mass fraction of water contained in the samples.

Thermogravimetric analysis, Annexe I, was performed in the temperature range of 30-700 °C under both inert ( $N_2$ ) and oxidative (air) atmospheres. The heating rates used were 2, 20, 40 and 100  $K min^{-1}$ . For VPE, WVPE, WRPE and BPE2 samples degradation occurred in a single step, regardless of the heating rate, in the range of 350-570 °C. On the other hand, for BPE1 sample indicated that degradation occurred in two consecutive steps from 200 to 550 °C.

TG analysis confirms that the overall reactions of all samples of PE and BPE2 also occur in single step, with maximum conversion observed at 440 °C, 490 °C, 500 °C and 520 °C to 2, 20, 40 and 100  $K min^{-1}$ , respectively. For BPE 1, two weight loss steps were observed, the first to 280 °C and the second close to 520 °C.

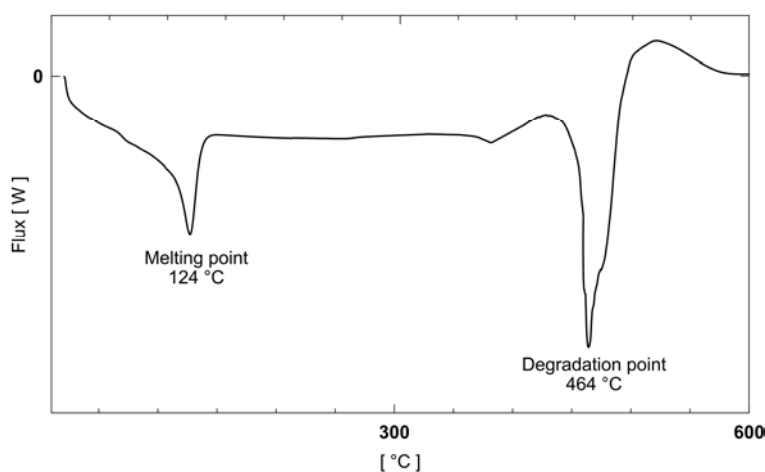
By TG curves was possible to establish the ash content. For the VPE the values were between 0.16 - 0.25 %, and for the WVPE and WRPE 0.2 to 1.05 %. For BPE1, the ash percentage was between 0.2 to 5.42 %. It is important note that for the BPE2 samples the residual mass is between 24.1 to 28.9 %, indicating that other chemicals compounds are present in their composition, ruling out the possibility of calculating the oxygen difference, and also the calculation of the LHV by Dulong's method.

The differential scanning calorimetry (DSC), Fig. 1, was carried out in the temperature range of 20-600 °C with heating rate of 10  $K min^{-1}$ , under an inert atmosphere. We observed two endothermic peaks. The first attributed to the polyethylene melting point around 124 °C, and the second around 464 °C, to the fission of the main polymeric molecule. Table 3 shows the other results for the DSC analysis.

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**Table 3.** The maximum temperature to the heat rate consumption.

Sample	Degradation temperatures obtained from DSC results (°C)	
	1st peak	2nd peak
VPE	124	464
WVPE	123	469
WRPE	124	475
BPE1	-	-
BPE2	-	-



**Figure 1.** Experimental results for Polyethylene Differential Scanning Calorimetry.

### 3. THERMAL CRACKING

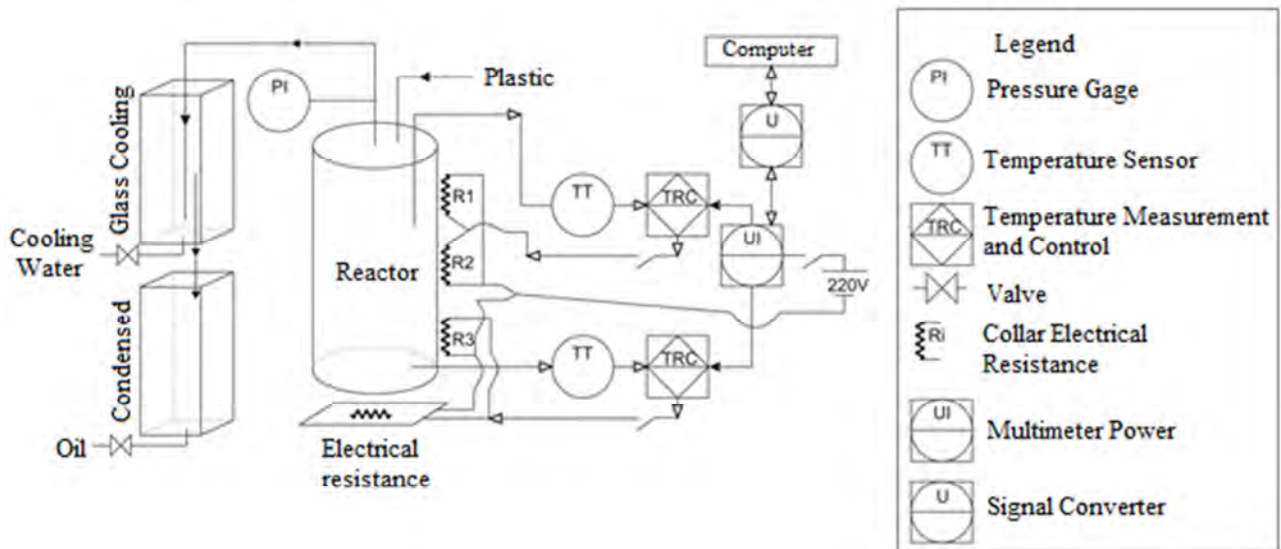
#### *By Electrical Resistance*

The thermal conversion of PE samples was performed by using the experimental cell of Duque and Schaeffer (2011), whose purpose was to investigate the possibility of recycling waste plastics by thermal degradation in order to transform them into nobler products and greater energetic value. Considering that plastic is derived directly from petroleum, the main idea was to recover a light oil that can be further refined into fuels.

The equipment, Fig. 2, is mainly divided into three parts: *control platform*, where the temperature is programmed and controlled; *reactor*, where the plastic is deposited and heated by electrical resistances, and *cooling the vessels*, one with liquid water another without just for condensate collection.

The experiments consist in heat to a temperature of 500 °C for a period of 2 hours, one kilogram of a waste recycled polyethylene resulting in approximately one liter of fuel. The oil, as well as the non-degraded residue remaining inside the reactor were collected and analyzed. Data collection for electricity consumption was conducted via computer with the help of multimeter power. Table 3 shows the experimental conditions and Tab. 4 laboratory analysis of oil obtained.

Table 5 shows, just to establish a comparison between the product obtained from thermal cracking with other common fuels a list HHV.



**Figure 2.** Experimental cell Schema with Electrical Resistance, micro-sampling device and data acquisition.

**Table 3. Experimental Conditions**

PARAMETER	MEASURE
Sample Weight [kg]	1,0
Working Temperature [°C]	500
Maximum Pressure Gage [kPa]	101,952
Test Time [h]	2,5
Average electric power consumption [Wh]	529,409
Fuel Volume Obtained [ml]	1.100
Residue remaining inside the reactor [kg]	0,053

**Table 4. Laboratory Analysis of Oil Obtained**

EXPERIMENT	RESULT	UNITS
Water Content	1,38	% (v/v)
Density at 20°C	0,7991	g/cm <sup>3</sup>
Degree API at 20°C	44,7	-
Kinematic Viscosity at 40°C	3,3565	mm <sup>2</sup> /s
Pour Point	37,0	°C
Salinity (NACL)	< 5,00	mg/L
Acidity Index	< 0,10	mgKOH/g
Sulfur Content	0,0061	% (m/m)
High heating value (HHV)	35.8	MJ/kg

**Table 5. High Heating Value of some fuels**

	FUEL	HHV (kJ/kg)
Petroleum Derived	Liquefied petroleum gas	49.9
	Kerosene	49.9
	Diesel	45.8
	Gasoline	43.9
Samples Collected	Oil obtained	35.8
	Residue remaining inside the reactor	27.9

### *Self-Sustaining Smoldering Combustion*

The reactor, Fig. 3, is finely instrumented. A group of eight in-line thermocouples 1.5 mm in diameter (T1, T2, T3, T4, T11, T12, T13, T14) are located at  $z = 0, 45, 90, 135$  and  $225, 270, 315, 360$  mm (from top to bottom of the reactor), making it possible to measure the temperature along the axis of the cell at different heights. A crown of six thermocouples, identical to the ones previously mentioned, makes it possible to measure the temperature over a horizontal cross section (at approximate middle height,  $z = 180$  mm) 6.5 mm away from the walls: this will reveal whether the combustion front progresses or not as a horizontal surface.

To start a uniform front across the whole surface of the bed, a sophisticated ignition device is used, called a Cone Calorimeter. A radiative flux is generated by a metal surface called a cone heater. The radiant cone heater temperature is adjusted to 570°C to impose a heat flux of 25 to 30  $KW m^{-2}$  over the top surface of the material. The cone heater has previously been calibrated. The radiative flux crosses a quartz porthole that ensures the sealing of the closure.

The experiments were run with an air flow rate of 0,676  $Kg h^{-1}$ . This corresponds to a Darcy velocity of 0.024  $m s^{-1}$  at 20.0°C, or 0.173  $m s^{-1}$  at 1000 °C. A mass of 615 g of mix 84/16 wt.% charcoal/WRPE was introduced in the cell.

The temperature values obtained at different locations within the bed for the experiment is illustrated in Fig. 4. It can be observed that the experiments using material containing large quantity of volatile matter is highly collapsing since the bed is consumed and collapses after oxidation of coal. Thus, these experiments show an instability in temperature profiles far superior to less collapsing experiments, requiring special techniques such as curve fitting and determination of the weighted average peak regions for the location of peak temperature for each thermocouple and calculating the velocity of the combustion front.

The propagating velocity of the combustion front is an increasing behavior. Al-Saffara *et al.* (2000) and Martins *et al.* (2010) found this trend. Torero and Fernandez-Pello (1996) also reports this acceleration during forward smolder of polyurethane foam in a forced air flow. However, in these unstable experiments, where the falling bed is constant due to the front passage, the instabilities cause changes in the velocity profiles resulting in the slow-down of the combustion front. It can be observed by increasing the distance between the average peaks of the thermocouples along the bed.

Furthermore, it can be seen that the crown peak emerged at different times, it indicates that the front propagates inclined, not horizontally. This may be due to instabilities in the ignition process or influence of heat losses on the wall. Another explanation may be the higher supply air in this region: the  $O_2$  density is higher on the walls, due to the lower temperature and the viscosity is smaller, also because the lower temperature.

On the horizontal cross section at mid-point of the height of the bed (Crown - T5 to T10) in all experiments can be seen that the peak temperatures were relatively lower than peaks along the axis of the cell. This can be attributed to significant heat loss through the walls of the cell. This observation indicates that in the literature experiments where the combustion front is observed in reactors without isolation of smallest diameter, the heat losses were probably much higher and significantly affect the structure of the combustion front. The reader can find detailed experimental and theoretical work about the effect of heat losses on opposed smoldering, and on critical conditions to achieve a self-sustaining combustion front in (Bar-Ilan *et al.*, 2004).

At the end of the experiment, the mass inside de combustion cell was insignificant, that means the self-sustaining front can easily crack thermally the polyethylene without additional energy except the one using during the ignition time.

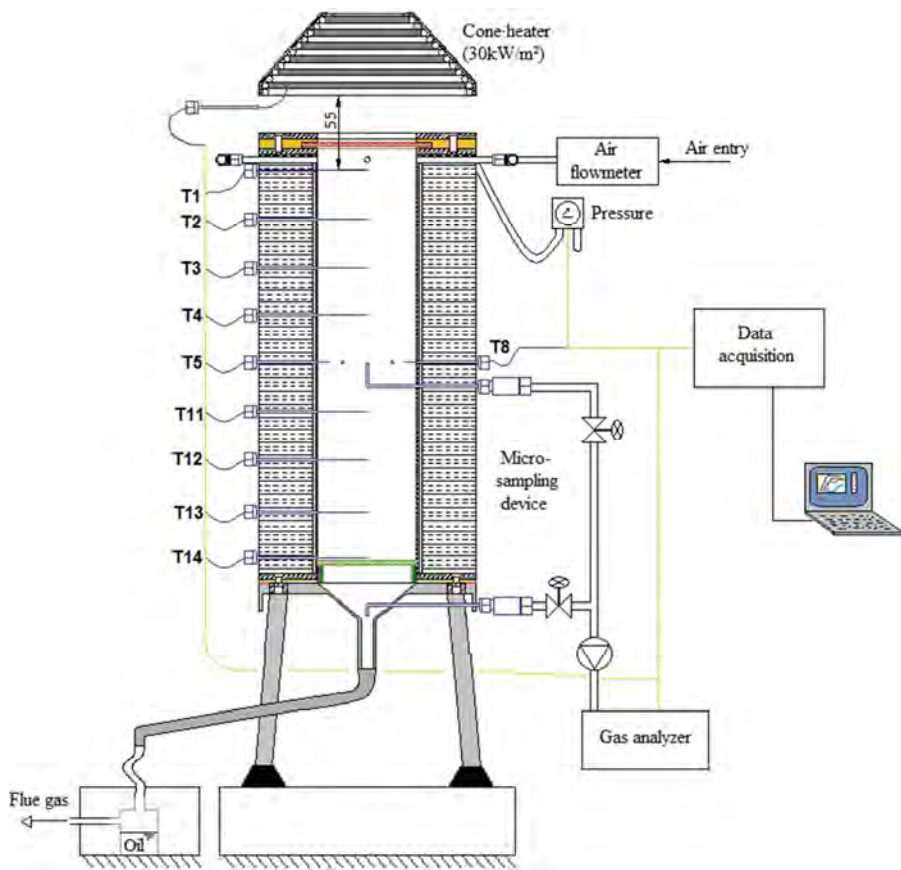


Figure 3. Combustion cell with micro-sampling device and data acquisition.

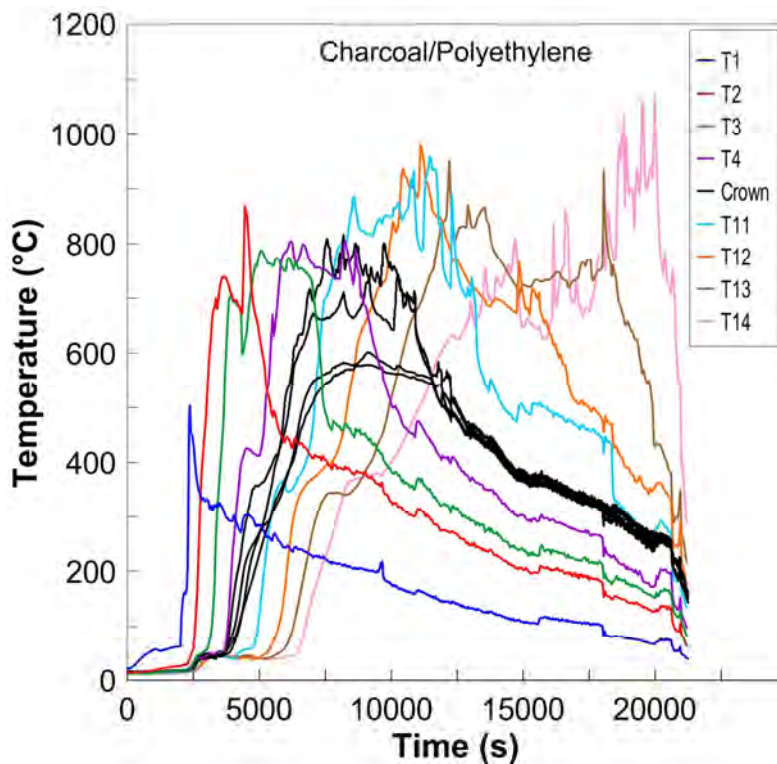


Figure 4. Values of temperature combustion of Charcoal / Polyethylene.

#### 4. CONCLUSION

A set of experimental procedure was carried out with success. The elemental analysis reveals the main presence of carbon and hydrogen in PE composition, except in the BPE samples that present other chemical compounds not characterized in the present study. By perform proximate analysis just moisture and ash fields where possible to be characterized, and confirmed by TG experiments where the total mass lost for Virgin Polyethylene, Waste Virgin Polyethylene, Waste Recycled Polyethylene and Biodegradable Polyethylene type 1 samples was an average 99.5 %, except to Biodegradable Polyethylene type 2 that lost just 72%.

The thermal conversion process deserves to be thoroughly investigated, since fuel oil, and also obtained residual product remaining in the reactor showed considerable calorific value. Another important observation is regarding the final reduction of mass of plastic waste, somewhere around 98.8%. May also be highlighted other properties presented by fuel oil, for example, low sulfur, acidity and salinity.

The experiments gave significant results. By using lower flow rates can get higher amount of energy available in the external walls of the reactor. The advance of the front is dependent on the supply of air. Their geometry tends to be non-uniform with slight inclinations and propagates faster in the periphery of the bed due to factors such as the ignition process instability or loss of heat through the walls, resulting in increased O<sub>2</sub> supply near the walls.

Despite the relatively large diameter of the cell and a good thermal insulation, heat losses are significant in relation to the heat generated by combustion.

The micro-sampling system identifies the gases inside the front and allows characterize it geometrically and chemically. There was a tendency to the emergence of two combustion systems in the cell. At the beginning there is *oxygen-limited* combustion and at the end *reaction-limited* combustion. The combustion system in all experiments produced a significant concentration of CO and H<sub>2</sub> indicating that gasification reactions occur.

The front velocity is an increasing function. However, in beds where constant collapses occur, due to their passage, it becomes decreasing. Falls in the bed, due to the formation of ashes, slow the front considerably and cause an instability in temperature profiles.

From the set temperature profiles, can be made the geometry of the drying, devolatilization and oxidation zone, if they exist, as well as others which happen to exist depending on the fuel used.

The pressure within the bed tends to increase due to the accumulation of condensate. When the condensates reach the end of the cell and are evacuated causes the pressure decreases.

Thus, by means of this new experimental device one-dimensional study of combustion front propagating to different waste fuels with air supply co-current and counter-current can be studied in a complete manner. Although focus is given to the co-current arrangement, for which the results were obtained, the cell also enable future studies in counter-current arrangement.

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**Annexe 1. Thermogravimetric analysis for Polyethylene.**

