

## AN EXPERIMENTAL STUDY OF RECOVERABLE PRODUCTS FROM WASTE TIRE PYROLYSIS

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### ABSTRACT

In this study, potential of useful products recovery was investigated from waste tires that completed the physical life by virtue of pyrolysis process. Disposal of waste tires, which is one of the global environmental problems, continues the presence thereof as a serious problem, especially in developed countries. Pyrolysis and gasification processes stand out due to making possible the disposal of waste tires and obtaining products that can be employed in energy recovery. Pyrolysis experiments were carried out in a fixed-bed-reactor with cyclone separator at various temperatures (300 °C, 400 °C, 500 °C, 600 °C and 700 °C) and nitrogen, employed as agent gas, was given batch and continuously during the process. In the study, the calorific values of the liquid and solid pyrolysis products were determined as 9117 kcal m<sup>-3</sup> and 8710 kcal kg<sup>-1</sup> respectively. When the results of the experimental studies were evaluated, synthesis gas rich in CH<sub>4</sub> and H<sub>2</sub> with a high calorific value of 4180 kcal m<sup>-3</sup> was achieved.

**Keywords:** waste to energy, waste derived fuel (WDF), thermal processes

### 1. Introduction

Since tires cannot break down in the nature on their own, they lead to severe environmental problems in case that they are not properly eliminated. Tires are the products of complex high technology and manufactured from a wide spectrum of materials and are made up of various components (Terry *et al.*, 2004). Almost 70-80% of the tires are made up of carbonated materials (Alkhatib *et al.*, 2015; Hita *et al.*, 2016) and potentially; these carbonated components can be recycled through thermochemical methods.

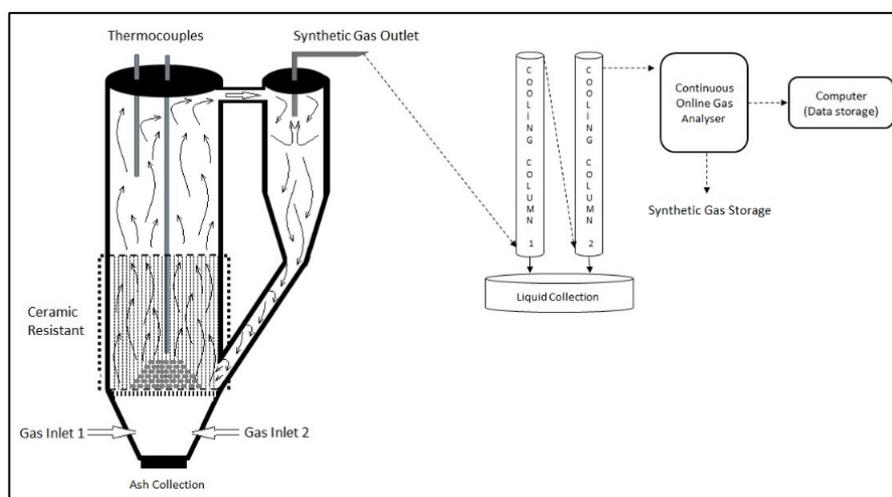
Annually, almost 1.4 billion tires are sold worldwide and almost the same numbers of tires are discarded (ETRMA, 2011). Approximately 3.4 and 4.6 million tons of discarded tires are estimated to be annually produced in Europe and the USA, respectively (Williams, 2013). The major methods used in waste tire management differ by according to economic costs and industrial usage (Rosendorfova *et al.*, 1998; Koçak and Alpaslan, 2011). Tires have 6000 kcal kg<sup>-1</sup> energy value and this figure equals to a high quality coal. For this reason, waste tires can be used as energy sources in cement kilns or in industrial establishments with similar burning units (Elbaba *et al.*, 2010). Additionally, a wide range of application area of the solid and liquid products obtained from the waste tires pyrolysis increases the interest in recycling methods (Ahoor and Zandi-Atashbar, 2014). Gases having high level of calorific value acquired at the end of pyrolysis are used in obtaining energy. Liquid gases, however, can be used as fuels in burning units (Williams, 2002). Char, which is obtained following pyrolysis, is used as active carbon or carbon black (Xiao *et al.*, 2008; Li *et al.*, 2010).

In general, pyrolysis process starts at between 350-550 °C and can increase up to 700 °C. Rubber pyrolysis procedures are generally performed between 250-500 °C, but some studies report that it might be up to 900 °C (Prakash and Karunaithi, 2008). Reactor type plays an important role in pyrolysis procedure. Reactor quality does not only determine the quality and quantity of the products that will be obtained but also the cost of the procedure. Mainly fixed and fluidized-bed reactors are preferred in pyrolysis procedure. Pyrolysis can be applied to raw materials such as coal and biomass, and is also an energy conversion process for solid wastes with highly varying quantities and qualities acquired from different sources (Bridgwater, 2003).

There are various studies conducted on pyrolysis in literature. While these studies particularly focus on gasification of coal (Lee *et al.*, 1998; Shackley *et al.*, 2006), a good number of studies on gasification and pyrolysis of waste tires and different materials is also available (Donatelli *et al.*, 2010; Rincon and Gomez 2012). When the studies performed on waste tires are examined, it is seen that authors generally investigated pyrolysis conditions (Gonzalez *et al.*, 2001; Leung *et al.*, 2002; Oyedun *et al.*, 2012) and obtaining recyclable pyrolysis products (Helleura *et al.*, 2001; Diez *et al.*, 2003; Laresgoiti *et al.*, 2004; Shah *et al.*, 2009). This study investigates the probability of obtaining product and products with economic value from the end-of-life tires using pyrolysis method within the scope of sustainable waste management. In pyrolysis experiments, fixed bed steel reactors with cyclone separator were used, and the impact of different experimental conditions on solid, liquid and gas products was investigated.

## 2. Materials and Method

In this study, end-of-life tires and tires classified as waste upon the removal from the cars were used as raw material. Pyrolysis experiments were conducted in steel reactor with cyclone separator and fixed bed (Figure 1). The reactor which was used in the experiments is a stainless steel with vertical shape and is 50 cm in length and 7 cm in diameter. Heat requirement of the system was met indirectly via ceramic resistance. Pure graphite and/or graphite-lead helical gaskets were used to prevent gas leak.



**Figure 1.** Schematic view of experimental setup

Two different pyrolysis procedures, continuous N<sub>2</sub> and intermittent N<sub>2</sub> were performed during the study. The pyrolysis procedure in which N<sub>2</sub> (continuous) was used as operating gas with 1 l min<sup>-1</sup> flow was conducted at 300, 400, 500, 600 and 700 °C. In the second part of the study, N<sub>2</sub> gas was used at the beginning of the experiment until O<sub>2</sub> was removed from the reactor. Then N<sub>2</sub> gas entry into the system was halted. This procedure, which is called intermittent procedure, was conducted at 400, 500, 600 and 700 °C separately. Thus, we were able to investigate the possible impact of nitrogen gas creating pressure within the system on the generation of pyrolysis products. The pyrolysis procedures conducted with

continuous N<sub>2</sub> were coded with “E” letter, and those conducted with intermittent N<sub>2</sub> were coded with “Z” letter. Experimental conditions of the trials are given in Table 1.

### 2.1. Analytical procedure

Synthetic gas measurements were performed with ABB-AO2020 model continuous gas analyser. CO, CO<sub>2</sub>, H<sub>2</sub>, CH<sub>4</sub>, and O<sub>2</sub> amounts within the synthetic gases that were generated during pyrolysis process were measured as volumetric percentage. Elemental analyses of the raw waste tire samples used in the study and the solid and liquid products obtained at the end of the experiments were performed. C, H, N and S amounts of the samples were determined through Truspec CHN-S elemental analysis. Calorific value of liquid products was analyzed with LECO AC500 bomb calorimeter. SEM imaging was performed with FEI Quanta 450 FEG-EDS model SEM-EDS device. Humidity content, ash content, burning loss and solid material content experiments were carried out in order to determine the physical and chemical properties of the waste rubber samples used in the experiments. These experiments were carried out according to Standard Methods. (SM 2540 B; SM2540 E). Approximate calorific values of producer gases were calculated according to H<sub>2</sub>, CO and CH<sub>4</sub> volumetric percentages (Ongen *et al.*, 2014; Bossel 2003; Pangaliyev 2014) determined by AO2020 online gas analyzer.

**Table 1.** Experimental conditions

Experiment	Particle Size (µm)	Sample (g)	Temperature (°C)	Agent	Flow rate (l min <sup>-1</sup> )
E1	500-1000	100	300	N <sub>2</sub>	1
E2	500-1000	100	400	N <sub>2</sub>	1
E3	500-1000	100	500	N <sub>2</sub>	1
E4	500-1000	100	600	N <sub>2</sub>	1
E5	500-1000	100	700	N <sub>2</sub>	1
Z1	500-1000	100	400	-	-
Z2	500-1000	100	500	-	-
Z3	500-1000	100	600	-	-
Z4	500-1000	100	700	-	-

## 3. Results and Discussion

In the first part of the study, we aimed to determine some physical and chemical properties of the waste tire to be used in the study. For this purpose, elemental analysis was performed, and humidity percentage, ash amount, burning loss, and solid material contents were ascertained. The results obtained from the elemental analysis of waste tire sample are given in Table 2.

**Table 2.** Elemental and proximate analysis results of raw waste tire

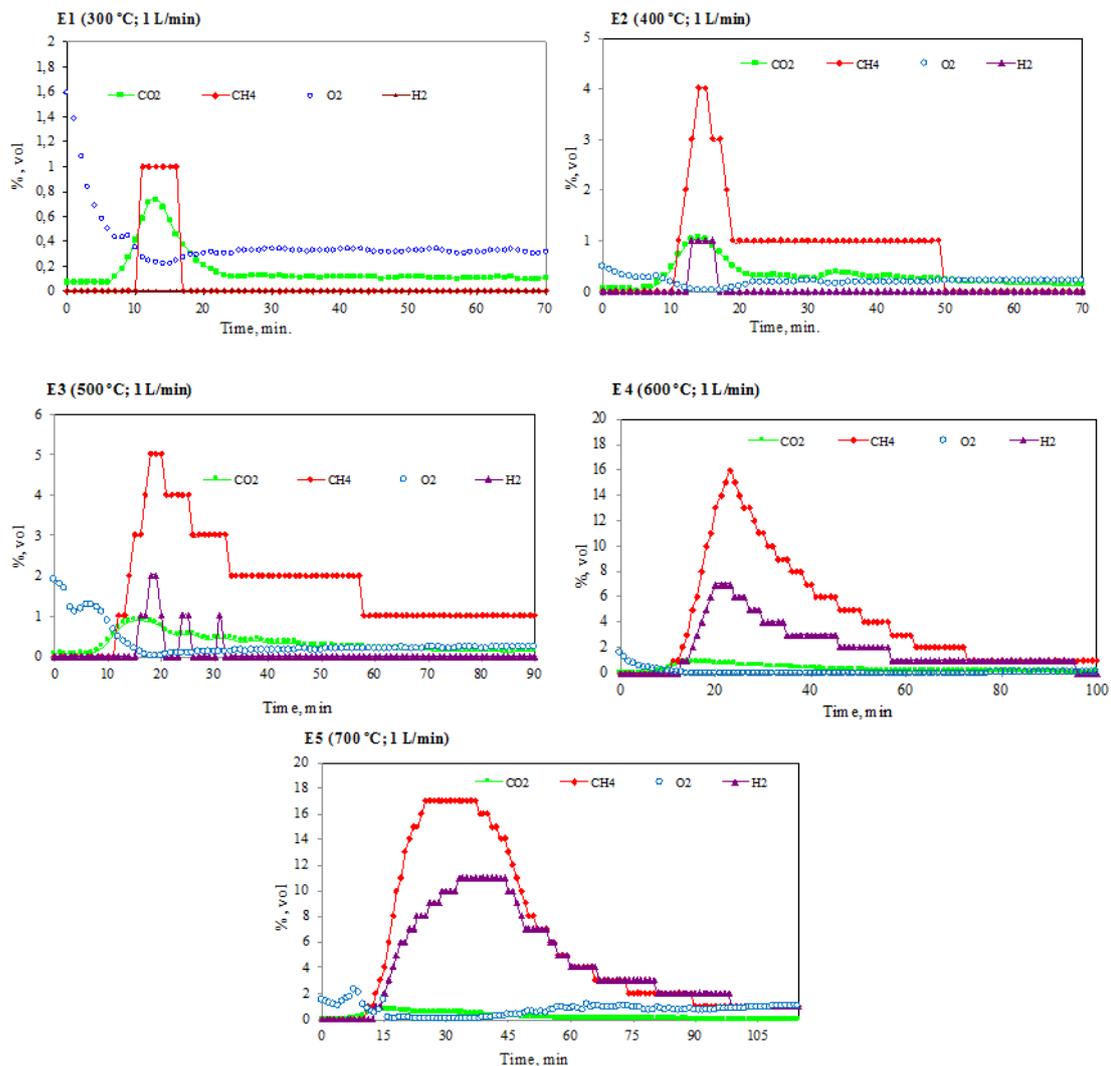
Ultimate Analysis, %mass					
C	H	N	S	O	Reference
80.4	8.7	0.3	1.6	9*	Present study
82.8	7.6	0.5	1.3	4.5	Dai <i>et al.</i> , 2001
86.7	8.1	0.4	1.4	1.3	Gonzalez <i>et al.</i> , 2001
Proximate Analysis, %mass					
Moisture	Ash	Combustion loss	Solid matter		
0.5	33.15	66.85	99.5	Present study	

\*calculated by difference

The fuel to be used for pyrolysis has a considerable effect on the efficiency of the product to be obtained. Gaseous and liquid products with high calorific value are mainly obtained from the fuels rich in carbon and hydrogen. The result of the elemental analysis given in Table 2 indicated that the sample contained 80.4% carbon and 8.7% hydrogen. These values are consistent with the literature, and show that the samples used in the study are suitable fuels for pyrolysis.

### 3.1. Synthesis Gas Analysis Results

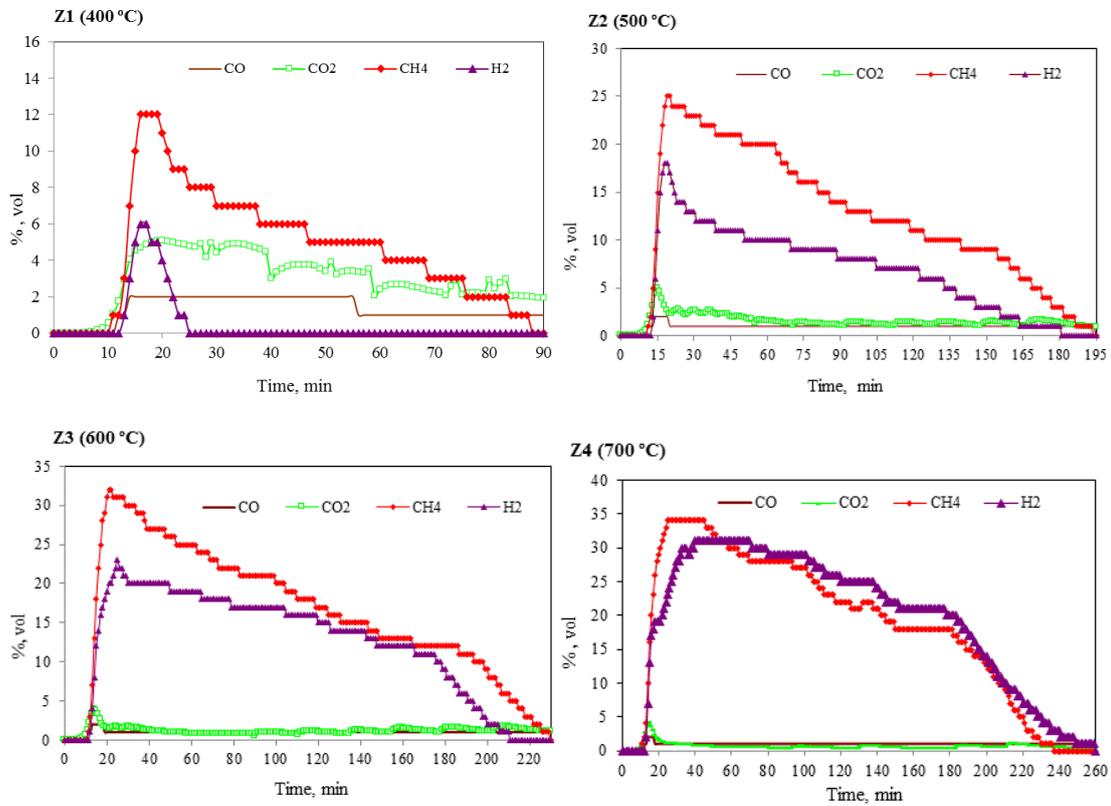
All synthesis gas values obtained in the experiments (E1, E2, E3, E4, E5) in which  $N_2$  was continuously fed are given in Figure 2. It was found out that  $CH_4$  and  $H_2$  gas percentages within the synthetic gas were low in the experiments conducted at 300 and 400 °C. It was observed that CO gas was not formed due to low temperature and that partial burning did not take place in the absence of oxygen during the experiments. Pyrolysis is also reported to be one of the reliable scenarios for the energy conversion from waste tires that produces syngas with very low CO and  $CO_2$ . During the experiments carried out at 600 and 700 °C that  $CH_4$  and  $H_2$  gas percentages within the synthetic gas are found higher in comparison to the experiments conducted at lower temperatures (400, 500 °C) (Figure 2). The highest  $CH_4$  (17%) and  $H_2$  (11%) levels were obtained in E5 experiment.



**Figure 2.** Synthesis gas content of continuous  $N_2$  pyrolysis experiments

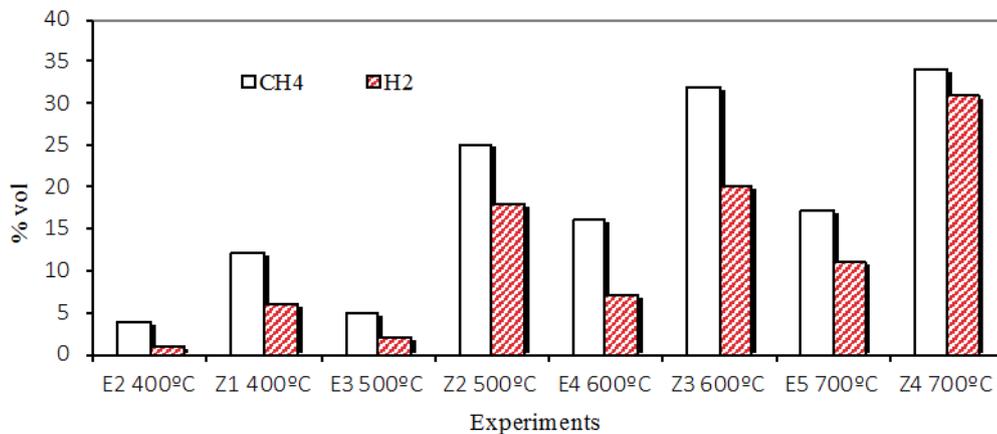
In the pyrolysis experiments (Z1, Z2) carried out at 400 and 500 °C with intermittent  $N_2$ ,  $CH_4$  increased from 12% to 25%. It was also found out that  $H_2$  gas percentage increased from 6% to 18% at 400 °C. In the experiments performed at 600 and 700 °C (Z3, Z4), it was determined that  $CH_4$  and  $H_2$  percentages increased considerably in comparison with the experiments conducted at low temperatures (400 and 500 °C). It was found that in all pyrolysis experiments conducted with intermittent  $N_2$ , the percentage of CO gas within synthesis gas is lower compared to  $CH_4$  and  $H_2$  gases. CO values observed at the experiments performed with intermittent  $N_2$  ranged from 0% – 2%. Gas conversion performance is expected to go up with the increase in temperature. An increase in pyrolysis temperature increased the yield of gaseous

products and decreased residue char production. Taking the data given in Figure 2 and Figure 3 into consideration, it was observed that CH<sub>4</sub> and H<sub>2</sub> gas percentages within synthesis gas obtained from the intermittent N<sub>2</sub> pyrolysis experiments were high.



**Figure 3.** Synthesis gas content of intermittent N<sub>2</sub> pyrolysis experiments

For this reason, maximum calorific value (4180 kcal m<sup>-3</sup>) of the synthetic gas in the experiments conducted with intermittent N<sub>2</sub> is higher than the maximum calorific value (1970 kcal m<sup>-3</sup>) of the synthetic gas in the experiments carried out with continuous N<sub>2</sub>. At pyrolysis phase, it was observed that continuous N<sub>2</sub> presence created a volumetrically limiting effect. Upon generating an environment free-of oxygen, it was seen that cutting N<sub>2</sub> entry into the system and increasing reaction temperatures enhanced synthesis gas efficiency. Besides, it was seen that the increase in operating temperature independent from the presence of N<sub>2</sub> gas increased CH<sub>4</sub> and H<sub>2</sub> gas percentages.



**Figure 4.** Highest values of CH<sub>4</sub> and H<sub>2</sub> gases obtained from pyrolysis

Figure 4 presents the highest values of CH<sub>4</sub> and H<sub>2</sub> gases obtained as a result of the continuous and intermittent pyrolysis carried at different temperatures. Calorific value of the synthesis gases obtained at the end of the experiments was calculated mathematically. The results obtained indicated that synthesis gases had high calorific value. The synthesis gas with the highest calorific value in pyrolysis experiments was obtained in experiment Z4 with 4180 kcal m<sup>-3</sup>.

### 3.2. Solid and Liquid Product Analysis

Liquid product (tar) and solid residue (char) collected at the end of pyrolysis experiments are given in Table 3. Elemental analysis of the solid and liquid products obtained as a result of continuous N<sub>2</sub> pyrolysis experiments was performed, and their calorific value was calculated. Table 4 represents elemental analysis and calorific value of liquid and solid products. In the study, while 90.2 g char was acquired in E1 experiment, 45.2 g char was obtained in E5. It was detected that carbon content mass of raw waste tire sample was 80.38%, whereas this value was 80.82 % in the pyrolysis performed at 300 °C. It was found that liquid products started to be acquired intensely at 500 °C and it continued up to 700 °C. If it is desired to properly complete the pyrolysis procedure carried out with the aim of enriching the carbon within the fuel at low temperatures, it should be taken into consideration that reaction time might be quite longer. According to the elemental analysis of the liquid product obtained at the end of the pyrolysis performed with continuous N<sub>2</sub>, demonstrated that it was found to be made up of a mixture of organic compounds, and contained carbon and hydrogen gases by weight (Table 4).

**Table 3.** Solid and liquid products obtained from pyrolysis

Experiments	Pyrolysis conditions	Raw waste tire (g)	Liquid product (g)	Solid products (g)
E1	continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 300 °C	100	0.4	90.2
E2	continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 400 °C	100	5.6	77.7
E3	continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 500 °C	100	6.5	67.2
E4	continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 600 °C	100	1.8	52.6
E5	continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 700 °C	100	21.3	45.2
Z1	intermittent N <sub>2</sub> ; 400 °C	100	0.5	80.9
Z2	intermittent N <sub>2</sub> ; 500 °C	100	8.1	62.3
Z3	intermittent N <sub>2</sub> ; 600 °C	100	5.3	53
Z4	intermittent N <sub>2</sub> ; 700 °C	100	9.8	49.2

As a liquid fuel, diesel fuel has 10256 kcal kg<sup>-1</sup> calorific value. Accordingly, the liquid product with 9117 kcal kg<sup>-1</sup> calorific value obtained at 700 C as a result of continuous N<sub>2</sub> pyrolysis can be regarded as an alternative fuel quite similar to the diesel fuel. The continuous N<sub>2</sub> and intermittent N<sub>2</sub> pyrolysis experiments revealed that liquid product amount obtained in pyrolysis experiments of high temperatures (>600 °C) is higher than the pyrolysis experiments of low temperatures (<600 °C). It was detected that heat increase in the pyrolysis experiments carried out with continuous N<sub>2</sub> and intermittent N<sub>2</sub> had an influence on the increase in the amount of liquid products (Table 3). When a comparison is made with regard to the calorific value of raw tire, it is observed that particularly mass form became rich in carbon at low temperatures. A relative decrease was observed in calorific values with the addition of the carbon within the fuel into the reactions with increasing heat. However, when all experiments are examined with multiple product perspective, it should not be disregarded that it is possible to benefit from each of the product type (mass, liquid, gas) both as an energy source and raw material. The limitations that arise from the fixed bed reactors used in the experiments are thought to be among the reasons why it is not possible to benefit from the carbon within the fuel in a more efficient way at high temperatures.

Waste tire granules used in our study were imaged with x80, x500, x1500 and x2500 percentage magnification using Scanning Electron Microscope (SEM) in order to monitor the pre and post-pyrolysis changes that occur in the solid product morphologies.

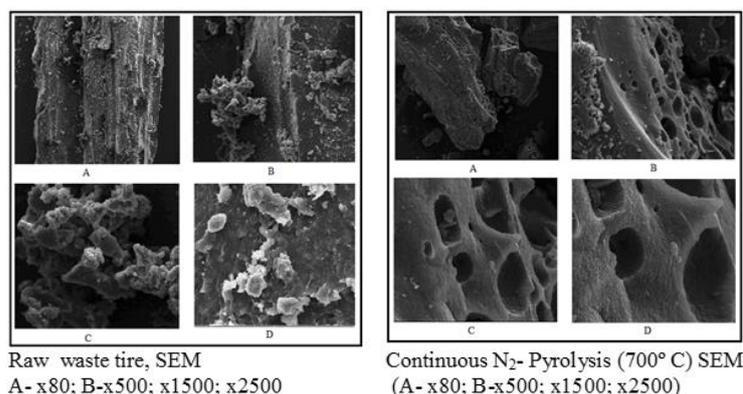
**Table 4.** Elemental analysis and calorific value of liquid and solid products from continuous N<sub>2</sub> pyrolysis experiments

Solid products					
	Elemental Analysis, weight %				Calorific Value (kcal kg <sup>-1</sup> )
	C	H	N	S	
Raw Tire	80.38	8.67	0.3	1.6	7883
E1 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 300 °C	80.82	1.34	9.65	1.14	8710
E2 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 400 °C	83.43	1.08	8.03	1.68	8254
E3 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 500 °C	77.85	5.51	1.28	1.92	7729
E4 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 600 °C	77.74	2.12	1.39	2.07	7363
E5 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 700 °C	74.53	4.27	1.66	1.95	6801

Liquid products					
	Elemental Analysis, weight %				Calorific Value (kcal kg <sup>-1</sup> )
	C	H	N	S	
E3 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 500 °C	64.61	10.08	0.30	0.72	9117
E4 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 600 °C	66.41	9.9	0.40	0.39	8751
E5 continuous N <sub>2</sub> -1 l min <sup>-1</sup> ; 700 °C	76.70	4.81	0.29	0.23	8140

Figure 5 presents the relevant data. SEM analysis indicated that pore size of the mass products obtained from continuous N<sub>2</sub> pyrolysis experiments (E5) of high temperatures (700 °C) increased with heat. Expansion in pore structure shows that solid products can be regarded as absorbent materials.

**Figure 5.** SEM micrographs of raw tire and post pyrolysis materials

#### 4. Conclusions

In this study, for the production and the characterization of the pyrolysis products, fixed-bed pyrolysis of waste tires was carried out. The results led to the following conclusions:

1. It was observed that liquid products began to be acquired intensely at 500 °C and continued to be acquired intensely up to 700 °C. The calorific values of the liquid products obtained varied between 8140 – 9117 kcal kg<sup>-1</sup>. 9117 kcal kg<sup>-1</sup> degree obtained in E3, where the highest calorific value was achieved, points out that this product can be used as an alternative energy source.
2. It was found that N<sub>2</sub> intermittent experiments pyrolysis lasted longer than N<sub>2</sub> continuous pyrolysis experiments. In pyrolysis experiments it was found that solid product amount decreases with temperature increase. While 90.2 g solid debris was obtained at 300 °C, it was observed that 45.2 g char left at 700 °C (Table 3). This case indicates that organic material moves away from the system with a higher degree as temperature increases.

3. Solid product obtained in the pyrolysis procedure carried out at 300 °C, had 8710 kcal kg<sup>-1</sup> calorific value. Considering that calorific value of the anthracite coal, one of the fossil fuels extensively used, is around 7700 kcal kg<sup>-1</sup> (TMMOB, 2010), this calorific value shows that a solid fuel with a higher energy than anthracite coal was obtained. It can be regarded that this calorific value might be increased to higher degrees with the extension of reaction time.
4. In addition to obtaining recyclable products with the pyrolysis of waste tires, it is also possible to achieve environmental gains. Management of waste tires with calorific methods ensure waste minimization in terms of both volume and mass to a great degree. At the end of the pyrolysis experiment performed at 700 °C (E5), it was seen that waste tires shrank 50-55 % in mass, and 55-60 % in volume (Table 3). It is believed that it will solve the space problem in storing the waste tires substantially.

This study provides valuable findings at selected experimental conditions for energy recovery, indicating that waste tires can be used as alternative energy sources. It is possible to achieve economic gain with the elimination of an environmental problem.

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