



## CO-PYROLYSIS OF PINUS SAWDUST AND COAL IN FIXED BED BENCH SCALE REACTOR

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**Abstract.** Nowadays, co-pyrolysis processes involving biomass and fossil fuels have attracted increasing interests for power generation. This paper presents an experimental study of a co-pyrolysis process of five blends composed by *Pinus elliottii* sp. sawdust residue and subbituminous coal from South Brazil (0, 15, 50, 85 and 100 wt. % of biomass in the mixture). Tests with replicate were conducted in a fixed bed bench scale reactor at temperatures ranging from 345 to 555 °C and heating rate of 10 °C.min<sup>-1</sup> in order to verify the influence of blend composition and reactor temperature on the pyrolysis process. The yield of the liquid products (oils), which showed two or three phases and small heating value, varied from 5.9 to 32.8 wt. % as material fed in the reactor changed from pure coal to pure biomass. On the other hand, the yield of the solid products (chars) was reduced from 87.4 wt. % (coal pyrolysis) to 35.4 wt. % (biomass pyrolysis). Both the highest liquid yield and highest heating value for the char fraction were obtained at 555 °C and 85 wt. % of sawdust in the mixture, showing the presence of synergetic effects on oil properties at such operational condition. These results can be used as preliminary data for further pyrolysis studies using these materials as fuel.

**Keywords:** Co-pyrolysis, pinus sawdust, subbituminous coal, fixed bed, biomass

### 1. INTRODUCTION

The thermal decomposition of biomass into chemicals compounds and derived fuels from pyrolysis processes is a promising technology to generate green energy. The process occurs in the absence of oxygen or in the presence of a significantly less oxygen amount than needed for complete combustion (Demirbas, 2011). Biochar is the highly carbonaceous solid product from pyrolysis of biomass, which can be used to improve the yield of various agricultural crops (Lee *et al.*, 2012) or can be transformed to activated carbon for adsorption processes (Pütün *et al.*, 2005) or even can be used as a more valuable conventional fuel (Apaydin-Varol *et al.*, 2007).

In the last decade, co-pyrolysis of biomass fuels and coal is being considered an effective alternative for reducing greenhouse emissions (Moghtaderi *et al.*, 2004). Simultaneously, the use of biomass residues can be a solution to landfill disposal, which has become very expensive in many countries (Collotet *et al.*, 1999). The use of carbon-neutral biomass added to coal is one of the most attractive options of reducing CO<sub>2</sub> emissions from power plants, once the carbon in biomass is generated from atmospheric CO<sub>2</sub> by photosynthesis (Weiland *et al.*, 2012).

Garcia-Pérez *et al.* (2001) results show that the co-pyrolysis of biomass and petroleum residues could potentially be a good solution to improve bio-oil properties. The high sulphur content and low pH of pyrolytic liquid fractions might be mitigated if both fuels are submitted to mixture before the pyrolysis process.

Cordero *et al.* (2004) confirmed the important beneficial effect of biomass on coal desulphurization (SO<sub>2</sub> emission reduction up to 80 %) upon thermal treatment. High quality solid fuels (char) were detected and, as a result, co-pyrolysis char presented higher commercial values than coal char, in agreement with the higher values of biomass char themselves.

Thermogravimetric analysis of coal/biomass blends were conducted by Vuthaluru (2004), who found that the highest reaction rate occurs when the coal/biomass blend ratio is 50:50. He also verified that no interactions occurred between coal and biomass during co-pyrolysis as the pyrolytic characteristics of coal/biomass blends followed those of the parent fuels in an additive manner.

The aim of this work is to verify the effects of temperature and blend ratio on the properties of co-pyrolysis products (solid and liquid fractions) in order to contribute towards knowledge regarding green conversion processes into enhancement fuels.

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## 2. MATERIALS, METHODS AND EXPERIMENTAL SYSTEM

### 2.1 Materials

Mineral coal type CE-4500 from Carbonífera Metropolitana (Criciúma, SC) and *Pinus elliottii* sp. sawdust from Cappelletta LTDA (Piracicaba, SP) were chosen for the present co-pyrolysis study. The air-dried biomass sample was sieved and only particles less than 3,360  $\mu\text{m}$  were selected to improve the particles homogenization inside the bed. The sawdust sample was grinded twice to reduce the particle size before pyrolysis tests. Particles size measurements shows a range between 31 and 1,410  $\mu\text{m}$  for coal and a range from 400 to 1,800  $\mu\text{m}$  for sawdust. Table 1 shows the fuels properties and methodologies used for fuel characterization.

Table1.Raw materials properties

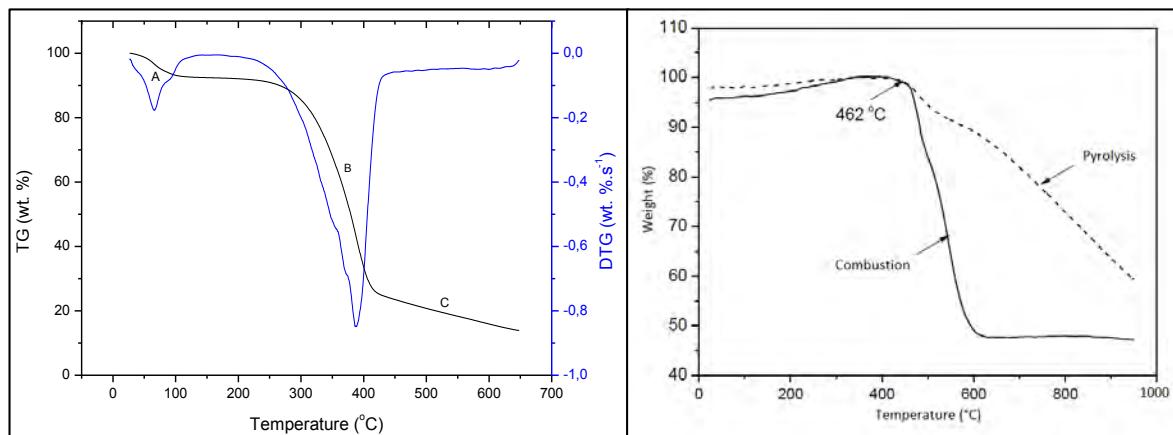
<b>Fuel Property</b> ( <i>Methodology</i> )	<i>Pinus elliottii</i> sp. sawdust	Mineral coal CE-4500
<i>Particle density (kg.m<sup>-3</sup>)</i> ( <i>Liquidpicnometry using N-heptane</i> )	1,214 $\pm$ 32	1,613 $\pm$ 26
<i>Real particle density (kg.m<sup>-3</sup>)</i> ( <i>Gáspicnometry using helium</i> )	1,510 $\pm$ 20	1,654 $\pm$ 1
<i>Particle size (<math>\mu\text{m}</math>)</i> ( <i>Sieving analysis</i> )	666 $\pm$ 3	461 $\pm$ 1
<i>Higher Heating Value (kJ.kg<sup>-1</sup>)</i> ( <i>BombCalorimeter</i> )	18,649 $\pm$ 90	20,295 $\pm$ 1
<i>Lower Heating Value(kJ.kg<sup>-1</sup>)</i> ( <i>Bomb Calorimeter</i> )	17,442 $\pm$ 101	19,532 $\pm$ 1
<i>Bulk density (kg.m<sup>-3</sup>)</i> ( <i>Mass and volume of the loosely packed bed</i> )	229 $\pm$ 6	813 $\pm$ 26
<i>Ultimate analysis(wt. %d.b.*)</i> Carbon Hydrogen Nitrogen Oxygen (difference) Sulphur ( <i>Perkin Elmer – series II 2400</i> )	47.87 $\pm$ 0.51 6.15 $\pm$ 0.27 0.14 $\pm$ 0.16 45.84 $\pm$ 0.33 n.d. <sup>***</sup>	51.70 $\pm$ 2.63 3.54 $\pm$ 0.24 1.09 $\pm$ 0.03 41.73 $\pm$ 2.81 1.94 $\pm$ 0.05
<i>Proximate analysis(wt. %w.b.**)</i> Moisture Volatile matter Fixed carbon Ash ( <i>ASTM E 870, 872, 1755, 1756 and ASTM D 3172 to 3175</i> )	9.13 $\pm$ 0.25 77.53 $\pm$ 0.10 13.17 $\pm$ 0.09 0.17 $\pm$ 0.15	1.65 $\pm$ 1.46 24.89 $\pm$ 0.85 36.14 $\pm$ 1.27 37.32 $\pm$ 2.11

\* drybasis; \*\* wet basis; \*\*\* notdetected

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## 2.2 Thermal analysis

Figure 1(a) and 1(b) shows the thermogravimetric and differential thermal analysis for sawdust and coal, respectively.



(a) (b)  
Figure 1. TGA/DTG curves for (a) sawdust; (b) coal (adapted from Crnkovic *et al.*, 2004 and Ávila, 2008)

This analysis was conducted in a Shimadzu 51H thermogravimetric analyzer. The initial sawdust sample was  $10.0 \text{ mg} \pm 0.5 \text{ mg}$  and the  $\text{N}_2$  flow rate was about  $100 \text{ mL} \cdot \text{min}^{-1}$  and heated from 40 (at  $10 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ ) up to  $650 \text{ }^\circ\text{C}$ . As can be seen in Fig. 1(a), the sawdust thermal decomposition presents three steps:

- (A) Moisture loss until slightly over  $100 \text{ }^\circ\text{C}$ ;
- (B) Devolatilization that starts near  $220 \text{ }^\circ\text{C}$  till  $410 \text{ }^\circ\text{C}$ ;
- (C) Final weight loss throughout analysis up to  $650 \text{ }^\circ\text{C}$ .

In the case of coal, however, the first weight loss starts at  $462 \text{ }^\circ\text{C}$ . Crnkovic *et al.* (2004) and Ávila (2008) concluded that it is not clear the degradation steps for coal.

Table 2 shows the measured structural analysis for the studied sawdust, where the weight fraction of hemicellulose, cellulose and lignin, the three structural components of biomass, is presented. Usually, the weight fraction of these components in lignocellulosic biomass are in the ranges 20-40 % (hemicellulose), 40-60% (cellulose) and 10-25 % (lignin) as discussed in Yang *et al.* (2007). The sawdust structural analysis was conducted in the Pulp and Paper Laboratory at Federal University of Viçosa, MG. To estimate cellulose and hemicellulose concentration, the procedure specified by Wallis *et al.* (1996) was followed. Lignin concentration in sawdust was determined using standard methods TAPPI UM 250 and TAPPI 222 om-98. Total extractives determination followed TAPPI T 264 cm-97.

Table 2. Weight fraction (%) of hemicellulose, cellulose, lignin and total in sawdust

Hemicellulose	Cellulose	Lignin	Extractives	Ash*
$19,6 \pm 0,1$	$41,9 \pm 0,1$	$28,6 \pm 0,4$	$8,3 \pm 0,2$	$0.8 \pm 0,1$

\*by difference

The different methodologies for ash content determination may explain the difference between the values from proximate analysis in Tab. 1 and the values from Tab. 2 regarding the sawdust ash content.

## 2.3 Experimental set-up

The co-pyrolysis was performed in a fixed bed reactor made in 305 AISI stainless steel as shown in Fig. 2. The internal diameter and height of the reactor are 15 mm and 220 mm, respectively, which is covered by four electrical belts, 800 W capacity each. A K-type thermocouple was inserted through the top of the reactor for temperature measurements ( $T_R$ ). The volatile gases from the pyrolysis reactor were conducted to the condenser water cooled ( $T_{w,i} \cong T_{w,o} \cong 22 \text{ }^\circ\text{C}$ ) by the pressure gradient in the system. The condensed vapor constitutes the pyrolytic liquid, which is collected during the test in a glass sample vial. The data acquisition system shows the reactor temperature during the test, allowing the heating process control by the operator using a voltage regulator (variac) connected to the electric heaters.

The non condensed gas was conducted to the stack where it was liberated to atmosphere.

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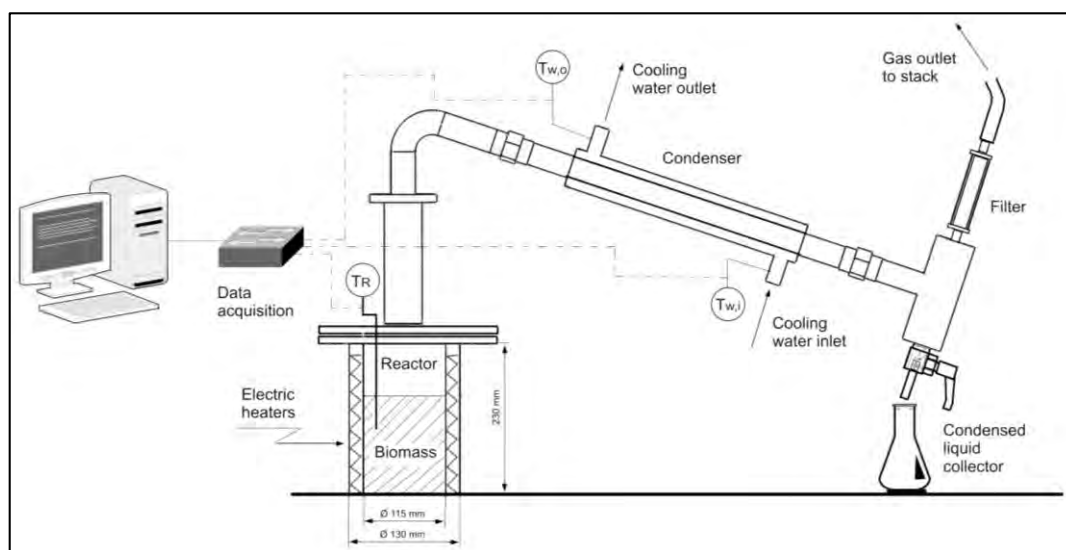


Figure 2. Schematic diagram of the pyrolysis fixed bed reactor

## 2.4 Procedure

Five blending ratios (weight of biomass to biomass-coal mixture,  $\tau$ ) were used, namely 0, 15, 50, 85 and 100%. Tests were conducted at a low heating rate for three different reactor temperatures (345, 450 and 555 °C).

The pyrolysis temperatures were achieved at roughly heating rates of 15 °C.min<sup>-1</sup>. For every experimental run, the reactor was feed with 250 g of fuel mixture and then closed. After that, it was heated up till the desired temperature. After this temperature was reached, the electrical resistances were controlled by a voltage regulator (0-120V) to maintain the reactor temperature constant, until the condensed liquid production stops (end of the test). At this point, the heaters were turned off. After the reactor has reached the room temperature, it was opened and the solid product was collected and weighted to obtain the solid yield ( $\eta_{sol}$ ). The liquid yield ( $\eta_{liq}$ ) was obtained by weighting the liquid collector and the condenser apparatus before and after each test. The gas yield ( $\eta_{gas}$ ) was obtained by difference from biomass weight added in the reactor (250 g).

Eleven different operational conditions were tested. Three blending ratios, for 345 and 555 °C reactor temperature, and five blending ratios for 450 °C reactor temperature. All tests were done in replica. The spent time in each test varied from 40 to 80 min depending on the reactor temperature.

A C2000 bomb calorimeter from IKA was selected to perform the high heating value of the solid and liquid pyrolysis products, which were measured twice for each material. A sample of 0.3 mg of each product was placed in the platinum crucible and inserted on bomb calorimeter container, which was fully injected with pure O<sub>2</sub> at 30 bar automatically.

## 3. RESULTS AND DISCUSSION

The solid, liquid and gas yields as well as the liquid and solid high heating values results, for each operational tested condition, are showed in Table 3.

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Table 3. Average products yields and standard deviation from fixed bed pyrolysis reactor

RUN	$\tau$ (wt. %)	Reactor temperature (°C)	$\eta_{sol}$	$\eta_{liq}$	$\eta_{gas}$	HHV	
			(%)			(kJ.kg <sup>-1</sup> )	
						solid	liquid
t <sub>1</sub>	15	345	84.5	5.9	9.6	18030	4576
t <sub>1R</sub>			83.9	5.8	10.3		
<b>Average</b>			<b>84.2 ± 0.5</b>	<b>5.9 ± 0.1</b>	<b>9.9 ± 0.5</b>		
t <sub>2</sub>	50	345	72.0	12.8	15.2	21258	4754
t <sub>2R</sub>			66.4	17.4	16.3		
<b>Average</b>			<b>69.2 ± 4.0</b>	<b>15.1 ± 3.2</b>	<b>15.7 ± 0.8</b>		
t <sub>3</sub>	85	345	52.8	22.4	24.8	23114	5925
t <sub>3R</sub>			59.7	18.8	21.4		
<b>Average</b>			<b>56.3 ± 4.9</b>	<b>20.6 ± 2.5</b>	<b>23.1 ± 2.4</b>		
t <sub>4</sub>	0	450	88.1	2.6	9.3	16379	18182
t <sub>4R</sub>			86.6	4.6	8.8		
<b>Average</b>			<b>87.4 ± 1.0</b>	<b>3.6 ± 1.4</b>	<b>9.0 ± 0.4</b>		
t <sub>5</sub>	15	450	79.2	8.0	12.8	17221	6538
t <sub>5R</sub>			79.9	8.2	11.9		
<b>Average</b>			<b>79.5 ± 0.5</b>	<b>8.1 ± 0.1</b>	<b>12.4 ± 0.7</b>		
t <sub>6</sub>	50	450	57.6	20.8	21.6	20239	7566
t <sub>6R</sub>			57.7	22.8	19.5		
<b>Average</b>			<b>57.6 ± 0.1</b>	<b>21.8 ± 1.4</b>	<b>20.6 ± 1.5</b>		
t <sub>7</sub>	85	450	42.4	28.8	28.8	27079	9297
t <sub>7R</sub>			42.2	31.6	26.2		
<b>Average</b>			<b>42.3 ± 0.1</b>	<b>30.2 ± 2.0</b>	<b>27.5 ± 1.8</b>		
t <sub>8</sub>	100	450	36.6	30.7	32.8	29247	8101
t <sub>8R</sub>			34.2	32.5	33.3		
<b>Average</b>			<b>35.4 ± 1.6</b>	<b>31.6 ± 1.3</b>	<b>33.0 ± 0.4</b>		
t <sub>9</sub>	15	555	80.8	10.0	9.2	16346	7381
t <sub>9R</sub>			74.6	11.1	14.4		
<b>Average</b>			<b>77.7 ± 4.4</b>	<b>10.5 ± 0.8</b>	<b>11.8 ± 3.6</b>		
t <sub>10</sub>	50	555	62.4	29.6	8.0	19743	7521
t <sub>10R</sub>			55.5	22.8	21.7		
<b>Average</b>			<b>58.9 ± 4.9</b>	<b>26.2 ± 4.8</b>	<b>14.9 ± 9.7</b>		
t <sub>11</sub>	85	555	36.0	33.6	30.4	30695	6481
t <sub>11R</sub>			37.6	32.0	30.4		
<b>Average</b>			<b>36.8 ± 1.1</b>	<b>32.8 ± 1.1</b>	<b>30.4 ± 0.1</b>		

As can be seen from Table 3, the liquid and gas yield increase as raise the biomass ratio, thus decreasing the solid yield.

In the present work, the maximum liquid yield (33 %) was reached for bed reactor temperature of 555 °C and 85 % biomass/coal ratio (t<sub>9</sub>). A similar liquid yield was obtained for the test using 100% biomass in the reactor at 450 °C.

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### 3.1 Products yields

The two studied factors (blending ratio and reactor temperature) affected the overall yields and the pyrolysis products properties.

Diez *et al.* (2012) calculated sawdust yields of 87 % for liquid due to degradation of the organic matter and it occurred at temperature less than 400 °C. The solid yield was low (15 wt. %). The present work shows some higher values for solid yields than those presented by Diez *et al.* (2012), which may be explained by the biomass properties and reactor type, as well as the cooling conditions for gases obtained from pyrolysis.

Some difficulties to clean the condenser after runs led to some large deviation on solid, liquid or gas yields. This problem affected, mainly, the gas yield as it depends on the solid and liquid measurements. High standard deviations were obtained for liquid yields due to the small liquid quantity produced in some tests, which increases the uncertainty of the result. Solid yields presented lower deviations once this final pyrolysis product was easier for collecting and weighing. Runs as  $t_1$ ,  $t_4$ ,  $t_9$  and  $t_{11}$  had indicated good variable control, once the standard deviations were less than 4.4 % in all products yields.

#### 3.1.1 Blending ratio effect

The solid, liquid and gas yields versus the blending ratio are illustrated in Figs. 3 to 5.

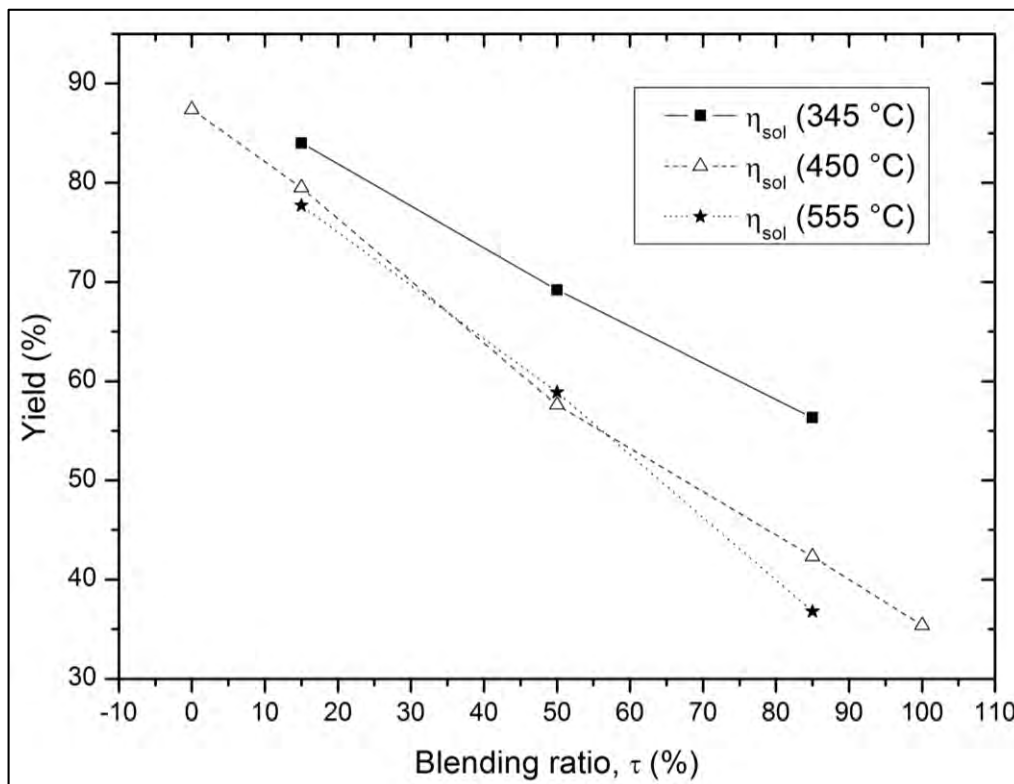


Figure 3. Solid yield versus blending ratio as a function of reactor temperature

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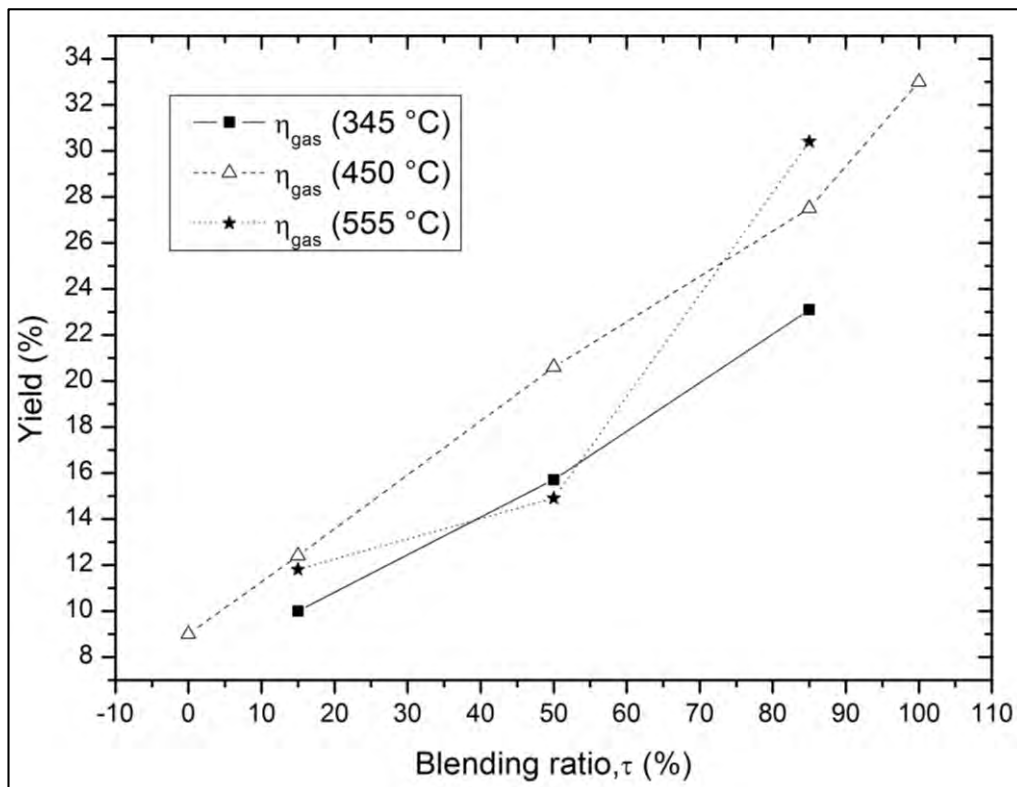


Figure 4. Gas yield versus blending ratio as a function of reactor temperature

The bed reactor temperature raised the liquid conversion (Fig. 5) for each biomass/coal ratio due to thermal degradation of fuels.

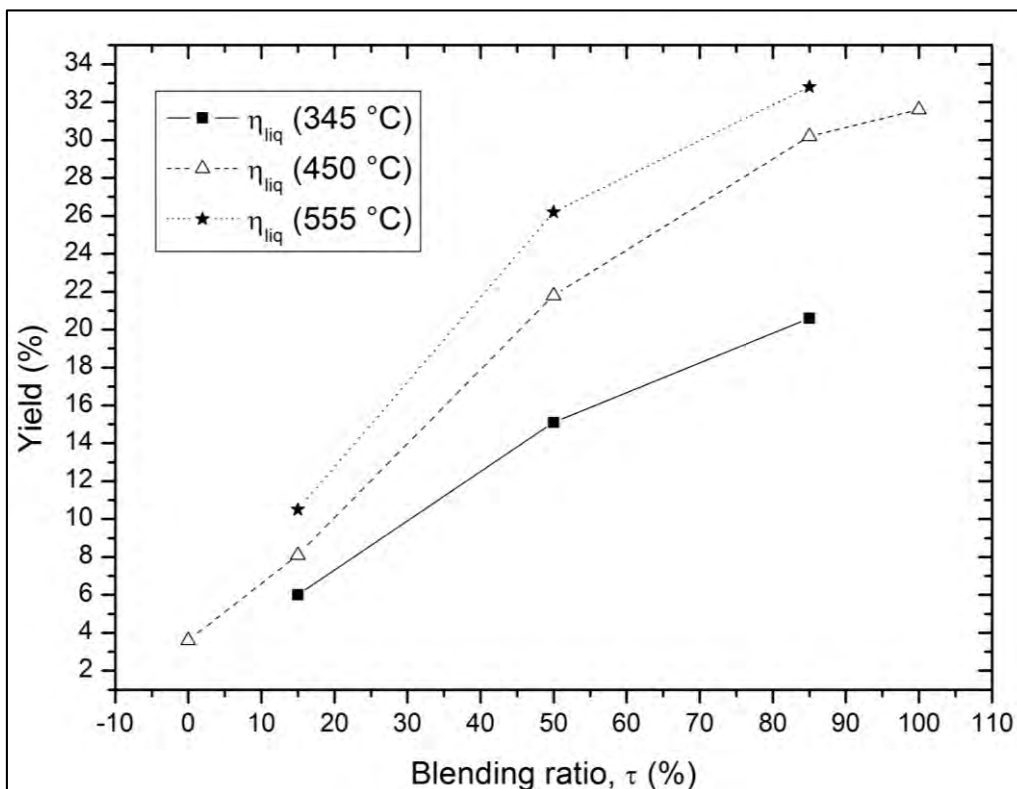


Figure 5. Liquid yield versus blending ratio as a function of reactor temperature

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From Fig. 3 it is noted the blending ratio promotes a diminution on solid yield due to more biomass devolatilization, mainly at temperatures around the 500 °C and thus increasing of the liquid yields (Figure 4). Zhang *et al.* (2010) explain that in order to maximize the solid production, low temperature and low heating rates are necessary. The amount of char generated during co-pyrolysis decreased with increasing biomass content in the blend, indicating absence of synergistic effects (Vuthaluru *et al.*, 2003).

Figure 5 shows the gas yield increased with increasing of blending ratio because the thermal decomposition of hemicellulose, cellulose and lignin, the three main biomass content. Perez *et al.* (2002) found the same trend in co-pyrolysis of petroleum residue and bagasse blends and they noted the increase concentration of petroleum residue increased the gas yield.

### 3.1.2 Reactor temperature effect

As visualized in Fig.3, the temperature parameter affects mainly major *pinus* sawdust blends. The reaction temperature reduced the solid yield due to component decomposition of biomass follows the increase of temperature (Yang *et al.*, 2007). Phan *et al.* (2008) concluded the pyrolysis gases increased with increasing the temperature.

Focusing on biomass and coal pyrolysis alone was noted that higher reaction temperatures leads to increase de gas yield and decrease the solid yield. Zhang *et al.* (2007) noted an increment on coal pyrolysis gas yield was only possible up to 600 °C. Diez *et al.* (2012) found the maximum temperature to liquid release was 576 °C for sawdust, which was significantly higher than the maximum temperature for the coal (491 °C). Phan *et al.* (2008) on gas characterization from pyrolysis of textile residues noted dramatic H<sub>2</sub> increase at higher temperatures.

### 3.2 High heating value of the liquid and solid products

Regarding the quality of the solids and liquids products as potential fuels, Figs. 6 and 7shows, respectively, the higher heating values obtained from measurements.

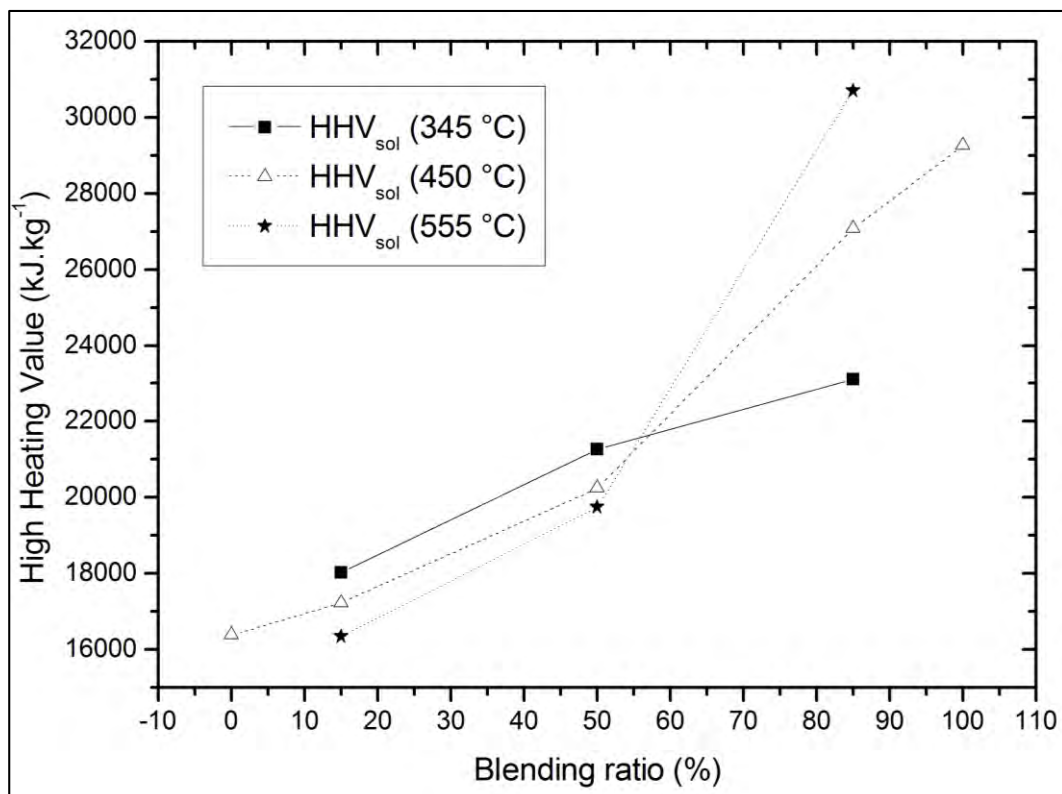


Figure 6. HHV solid products versus blending ratio as a function of reactor temperature



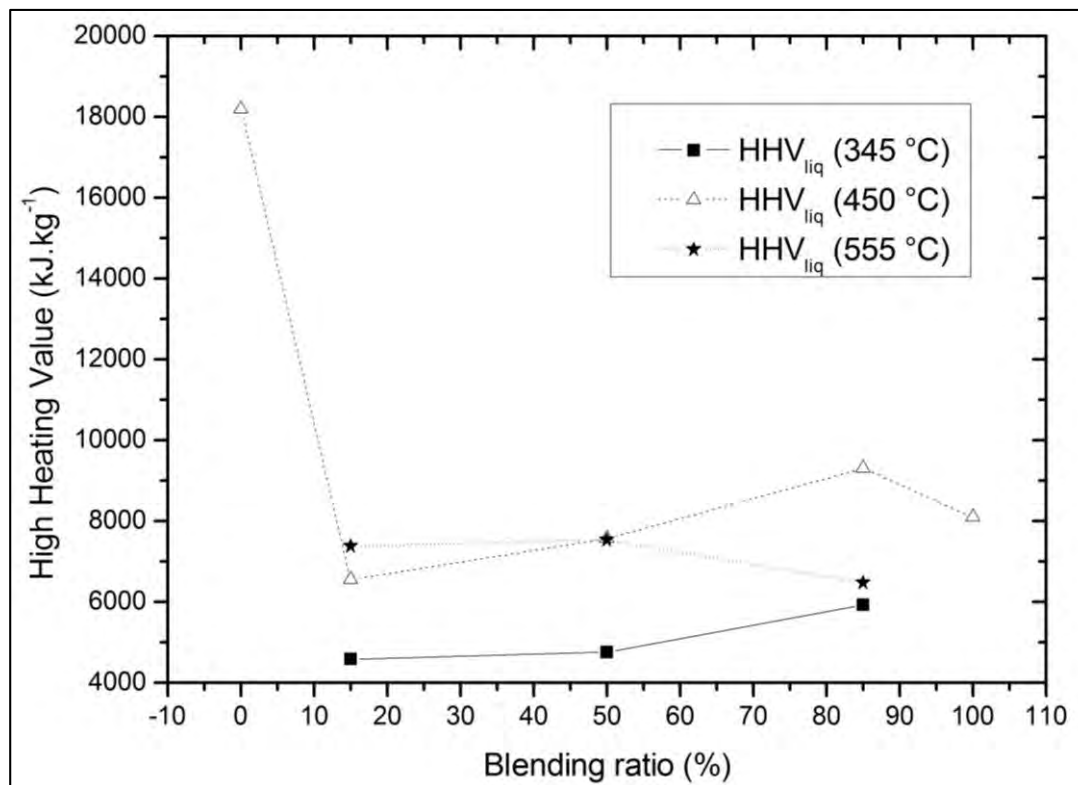


Figure 7. HHV liquid products versus blending ratio as a function of reactor temperature

Results confirmed that increase temperature raised the HHV, but not for run  $t_9$ , where the heating value was diminished. Figure 6 suggests that increasing the reaction temperatures promotes a higher HHV for solid pyrolysis product only for raw material ratio of 85 % sawdust in fuel mixture. For biomass proportion of 15 and 50 %, when the temperature increased, the heating value decreased slightly. On the other hand,  $t_{11}$  showed the highest heating value of solids. Mohan *et al.* (2005) showed HHV of liquid from wood pyrolysis between 16 and 19 MJ/kg with water content of 15 – 30 wt. %. The values measured were lower than those presented by Mohan *et al.* because all liquid fractions were collected throughout run, probably with water content higher than 30 % wt, justifying the lower values.

The highest solid HHV was obtained for 85 % wt. of biomass at 555 °C ( $t_{11}$ ), where coal was thermally degraded enough to increase the HHV. Cordero *et al.* (2004) found HHV of 26,263 and 34,288 kJ/kg for pure raw materials, coal and pine sawdust, respectively. For co-pyrolysis of 1:2 coal:pine sawdust blend, these authors found result HHV of 29,845 kJ/kg. Comparing it with values from runs  $t_2$ ,  $t_6$ ,  $t_{10}$ , there were lower than those found by researches. The explanation might be based on the difference temperature that Cordero used (600 °C), since the maximum fixed bed reactor temperature reached 555 °C.

#### 4. CONCLUSIONS

The test rig behavior was good for solid product evaluation with standard deviation expected. In some runs, the condenser generated large deviations on liquid yields. The slow pyrolysis was beneficial to generate solid product of pyrolysis.

The blending ratio and temperature parameters affected the pyrolysis products yields. As higher the biomass weight percent in fuel mixture and temperature reactor, more liquid and gas yields were obtained, with the opposite behavior for solid yields. Regarding the solid fraction's higher heating value, the parameters were analyzed led to increase all of them due to more decomposition of biomass components and volatile matter because increasing of biomass concentration. Conclusions are difficult to summarized about HHV of liquid, because of a clear tendency was not evident.

The blends of the two fuels led to increase the heating value when the biomass proportion increased in the fuel mixture, mainly in solid fraction, but this trend was not observed for the HHV of liquid, as can be seen on liquid fraction at 555 °C. Maybe the two fuels started combined reactions due to coal devolatilization, which affected the sawdust degradation at 555 °C, generating more water compounds, decreasing the HHV of liquid fraction for higher biomass blending ratio.

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## 5. ACKNOWLEDGEMENTS

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