

Vacuum Pyrolysis of Brazilian coal, peat and biomass – 1. Preliminary results on characterization of feedstock, solid residues and conversion rates

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ABSTRACT

Pyrolysis of solid fuels such as coal, peat and biomass is one of the principal technologies for the generation of liquid and gaseous products from these materials, as well as for the generation of a solid residue (char). The data presented here come from a recently installed bench-scale vacuum pyrolysis unit at the Instituto de Geociências, UFRGS, manufactured at Pyrovac Inc, Canada. Until now 6 Brazilian coal samples were tested along with 10 biomass and two peat samples. Feedstock samples and their respective solid residues (chars) obtained from the vacuum pyrolysis experiments were characterized by a number of analytical methods including proximate and ultimate analyses, determination of gross calorific value, petrographic analyses, reactivity to CO₂ and Raman spectroscopy and results are discussed in this paper. The preliminary results of this study suggest that among the coal samples investigated major conversion to liquids and gases (29.7-33.2 %) occurs in the high volatile bituminous coals from Santa Catarina, whereas in the biomass samples Mamona (Castor Beans), wood chips (Eucalyptus), wooden bars (pinus) and signal grass have all conversion rates > 60 %. The conversion rates for the peat samples varied between 32.5 and 46.6 %.

Key-words: Vacuum Pyrolysis, Brazilian Coal, Peat, Biomass, Conversion Characterization

RESUMO

A pirólise de combustíveis sólidos como carvão, turfa e biomassa é uma das principais tecnologias para a geração de produtos líquidos e gasosos a partir destes materiais, bem como para a geração de um resíduo sólido (char). Os dados aqui apresentados provêm de uma unidade de pirólise a vácuo de bancada instalada recentemente no Instituto de Geociências da UFRGS, fabricada na Pyrovac Inc, Canadá. Até o momento foram testadas 6 amostras de carvão, 10 amostras de biomassa e duas de turfa de origem brasileira. As amostras de matéria-prima e seus respectivos resíduos sólidos obtidos dos experimentos de pirólise a vácuo foram caracterizados por uma série de métodos analíticos incluindo análise imediata e elementar, determinação do poder calorífico superior, análises petrográficas, testes de reatividade ao CO₂ e Espectroscopia Raman. Os resultados são discutidos neste artigo. Os resultados preliminares deste estudo sugerem que, entre as amostras de carvão investigadas, ocorreu maior conversão em líquidos e gases (29,7-33,2%) nos carvões betuminosos altamente voláteis de Santa Catarina, enquanto nas amostras de biomassa Mamona madeira de eucalipto, barras de madeira (pinus) e grama de sinal têm taxas de conversão > 60%. As taxas de conversão para as amostras de turfa variaram entre 32,5 e 46,6%.

Palavra-chave: Pirólise a vácuo, carvão brasileiro, turfa, biomassa, caracterização da conversão

1 INTRODUCTION

Pyrolysis of solid fuels such as coal, peat and biomass is one of the principal technologies for the generation of liquid and gaseous products from these materials (Hebert et al, 1987, Kalkreuth et al, 1986a, 1986b, 1989a, 1989b, 1993; Roy et al, 1985, 1993, 1998; Pakdel et al, 1999), as well as for the

generation of a solid residue (char). The data presented here come from a recently installed bench-scale vacuum pyrolysis unit at the Instituto de Geociências, UFRGS. The equipment was manufactured by Pyrovac Inc, Canada.

The overall objective of this study is to determine the conversion characteristics of selected

coal, peat and biomass samples, as well as to test blends of peat/coal and coal/biomass samples, and to determine the properties of the solid residues and organic liquids derived from the experiments.

Until now 6 representative coal samples were tested. They were collected from open and underground mines in the states of Rio Grande do Sul, Santa Catarina and Paraná. The 10 biomass samples tested so far include some of the more common biomass types grown in Brazil, namely sugar cane, Eucalyptus tree, Signal Grass and Rice husks. The 2 peat samples tested were collected by shallow drilling from a peat deposit at Águas Claras, RS. The coal, biomass and peat feedstocks as well as their respective residues (chars) were analyzed by a number of chemical and physical parameter such as proximate and ultimate analyses, gross calorific values, vitrinite reflectances, degree of reactivity and Raman Spectroscopy.

2 VACUUM PYROLYSIS EXPERIMENTS

The experimental setup is shown in Figure 1.

Figure 1- Experimental Setup of the vacuum pyrolysis experiments, showing heating chamber to the left, one metallic trap, 3 glass traps to the front and the control panel to the back



The unit has a maximum capacity for testing 400 g of sample material, heated to a maximum of 550 °C at a heating rate of 10 °C/min and a total pressure regime of < 1.0 kPa. The sample is kept at maximum temperature for 2 hours. An image of biomass prior and after vacuum pyrolysis along with the derived liquid products is given in Figure 2.

The liquid products were condensed during the experiments in 4 traps connected in series, applying cooling temperatures of 0 °C (metallic trap) and -74 °C for the three remaining traps. The solid residue

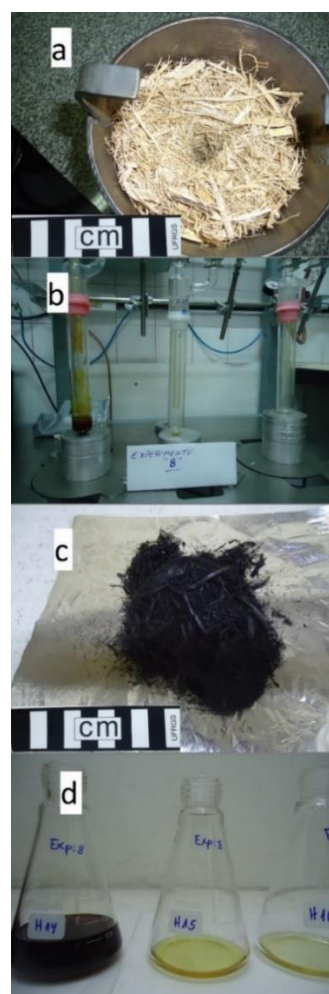
(char) was removed from the reactor after cooling down to close to room temperature.

3 ANALYTICAL METHODS

3.1 PROXIMATE AND ULTIMATE ANALYSES, GROSS CALORIFIC VALUE, PETROGRAPHIC ANALYSES

Ash yields, moisture, volatile matter and fixed carbon contents were determined by proximate analyses following ASTM Standard D3172 (ASTM, 1991). C, H, N and S contents were determined by ultimate analyses following ASTM Standard D3176 (ASTM, 1991) and Gross Calorific values was determined following ASTM standard test method 2015-91 (ASTM, 1991). Vitrinite Reflectance Measurements and Maceral Analyses were carried out conforming standardized procedures (Bustin et al., 1989).

Figura 2 - Images from experiment 8: (a) Sample of sugar cane bagasse prior to vacuum pyrolysis; (b) "traps" with condensed liquid products; (c) solid residue (char) derived from sugar cane bagasse; (d) image of liquid products removed from the traps by a mixture of dichloromethane and methanol.



3.2 REACTIVITY TO CO₂

Reactivity of the feedstock and solid residue was isothermally measured in a thermobalance at 1000 °C under CO₂ after heating the samples in N₂ flow (100 mL/min and 20 °C/min heating rate). The mean reaction rate was calculated using the expression $RCO_2 = 1/\%_{in} (d\%/dt)$, with $\%_{in}$ being the initial percentage of sample and $d\%/dt$ the derivative of instantaneous sample percentage.

3.3 RAMAN SPECTROSCOPY

Micro-Raman spectroscopy was performed using a 50x objective to focus a 632,8 nm laser on a 2 µm spot on the surface of the powdered and compacted sample. Laser power was reduced to avoid sample damage. Acquisition time was adapted to the response to laser excitation, in order to keep the intensity within the counting limits of the LN-cooled CCD-strip. The spectra were fitted with a polynomial baseline and two Lorentzian peaks (Schmidt et al, 2017), one for the graphite peak (G-peak) at approximately 1580 cm⁻¹, and the other related to the disorder in the carbon structure (D-peak) at approximately 1340 cm⁻¹.

4 RESULTS AND DISCUSSION

Results from petrographical and chemical analyses of feedstock and solid residues (chars) are summarized in Tables 1 and 2.

4.1 CHARACTERIZATION OF COAL FEEDSTOCK AND SOLID RESIDUES (CHARS)

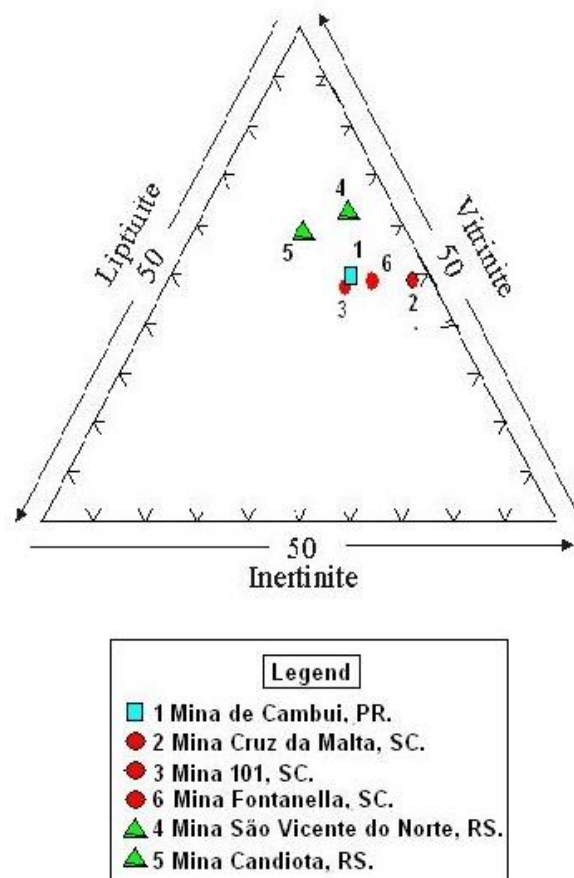
Results from analyses of feed coals (Table 1) show a rank ranging from subbituminous to high volatile A bituminous (Ro between 0.44 to 0.88 %) except for one sample, which has been partially altered by contact with a volcanic intrusion and shows a bimodal distribution of vitrinite reflectances (Ro 0.93 and 1.18 %). Results from maceral analyses indicate the predominance of macerals of the vitrinite group (Figure 3), with values > 60 vol% in the samples from Rio Grande do Sul, followed by inertinite macerals ranging from 20.3 to 47.1 Vol%. Liptinite group macerals occur in less quantities ranging from 2.0 – 18.4 Vol% (Figure 3).

Ash yields in the coals range between 11.9 and 46 wt.% (Table 1), and gross calorific values range between 2873 and 6484 cal/g).

The solid residues (chars) derived from the coals are characterized by a relative increase of ash yields (17.3-61.8 wt.%), when compared to the feedstock (Table 1) and the high vitrinite

reflectances (2.37 – 10.64 % Ro) are an indication of severe thermal alteration of the organic matter.

Figure- 3: Ternary diagram showing maceral group distribution in the coals (mineral matter free basis)



4.2 CHARACTERIZATION OF BIOMASS FEEDSTOCK AND SOLID RESIDUES (CHARS)

Results from analyses of biomass and their respective solid residues (chars) rather indicate low ash yields (0.6 – 12.3 wt.%) in the feedstock (Table 2) except for the rice husk sample having an ash yield of 21.1 wt.%, whereas Gross calorific values range from 3155 to 4688 cal/g. Analyses of the biomass solid residues (chars) show a significant increase in ash yields (1.6 – 52.2 wt.%) and Gross calorific values (3832 – 8031 cal/g), when compared to feedstock (Table 2).

Table 1: Chemical and physical (vitrinite reflectance) analyses of feed coals and their respective solid residues (chars)

Company	Mine	Exp.	Sample	Ash	VM	Moist.	FC	VM (%)	FC (%)	C	H	N	S	O	CV(cal/g)	Rrandom
				(%)	(%)	(%)	(%)	d.a.f.	d.a.f.							(%)
Cambui-PR	Cambui	3	16-026	11,9	31,2	5,4	51,5	37,7	62,3	65,7	4,7	1,2	3,3	13,2	6484	0,54
Rio Deserto-SC	Cruz de Malta*	4	16-027	42,7	20,6	0,4	36,4	36,1	63,9	49,6	2,8	0,8	2,5	1,6	4786	1,09***
Metropolitana-SC	Fontanella*	16	16-113	40,0	20,8	0,4	38,8	34,9	65,1	46,9	1,4	0,6	1,8	9,3	5006	0,84
Rio Deserto-SC	Mina 101**	5	16-028	37,8	23,4	0,3	38,6	37,8	62,2	52,8	3,4	0,9	1,0	4,0	5106	0,88
CRM-RS	São Vicente do Norte	6	16-029	33,1	25,1	7,6	34,2	42,4	57,6	45,3	3,9	0,7	0,8	16,3	4348	0,49
CRM-RS	Candiota	7	16-030	46,9	22,2	9,7	21,3	51,1	48,9	31,2	3,2	0,5	1,0	17,3	2873	0,44
Solid Residues- Results from proximate and elemental analyses and calorific value determination, Rrandom																
Company	Mine	Exp.	Sample	Ash	VM	Moist.	FC	VM (%)	FC (%)	C	H	N	S	O	CV(cal/g)	Rrandom
				(%)	(%)	(%)	(%)	d.a.f.	d.a.f.							(%)
Cambui-PR	Cambui	3	16-026 -R	17,3	10,3	1,3	71,1	12,6	87,4	73,7	2,6	1,5	2,7	2,2	6685	7,55
Rio Deserto-SC	Cruz de Malta*	4	16-027 -R	50,4	1,5	0,4	47,7	3,1	96,9	44,7	1,5	0,8	2,1	0,5	3988	10,64
Metropolitana-SC	Fontanella*	16	16-113-R	49,2	6,5	0,8	53,5	10,8	89,2	n/a	n/a	n/a	n/a	n/a	4154	n/a
Rio Deserto-SC	Mina 101**	5	16-028 -R	50,1	4,6	1,9	43,4	9,6	90,4	47,7	1,6	0,9	1,1	n/a	4090	8,81
CRM-RS	São Vicente do Norte	6	16-029 -R	40,5	9,4	0,5	49,6	15,9	84,1	46,3	1,6	0,8	0,6	10,2	4481	2,45
CRM-RS	Candiota	7	16-030 -R	61,8	6,4	1,0	30,8	17,2	82,8	33,4	1,2	0,5	1,3	1,8	3076	2,37

* coal seam Bonito

** coal seam Barro Branco

*** two vitrinite populations, with modes of 0,93 and 1,18 % Rrandom respectively

n/a = not analyzed

Table 2: Chemical analyses of biomass and peat feedstock and their respective solid residues (char)

Biomass and Peat - Results of proximate and elemental analyses and calorific value determination															
Company	Biomass	Exp.	Sample	Ash	VM	Moist.	FC	VM (%)	FC (%)	C	H	N	S	O	CV(cal/g)
				(%)	(%)	(%)	(%)	d.a.f.	d.a.f.						
Eco Fogo	Sugar Cane Bagasse	8	16-045	3,5	83,7	2,6	10,2	89,1	10,9	44,9	6,5	0,2	0,2	44,8	4092
Eco Fogo	Rice Husks	9	16-046	21,1	58,6	8,8	11,5	83,6	16,4	35,2	5,5	0,3	0,1	37,8	3155
Eco Fogo	Saw Dust	10	16-047	0,6	81,1	4,0	14,3	85,0	15,0	47,6	6,8	0,1	0,1	44,7	4360
Eco Fogo	wood chips , eucalyptus	11	16-048	1,3	79,8	5,6	13,3	85,7	14,3	47,0	6,8	0,1	0,1	44,8	4282
Eco Fogo	Eucalyptus Bark	14	16-111	4,9	20,9	4,5	69,7	23,0	77,0	n/a	n/a	n/a	n/a	n/a	4004
Eco Fogo	Eucalyptus Leaves	15	16-112	4,4	16,7	7,0	71,9	18,8	81,2	n/a	n/a	n/a	n/a	n/a	4688
Eco Fogo	Mamona/Castor Bean/Ricinus Communis L.	17	16-114	9,9	13,3	9,3	67,5	16,5	83,5	35,2	5,9	3,6	0,4	45	3720
Eco Fogo	Capim Camarão/Lollipop Plant	18	16-115	12,3	27	0,8	59,9	31,1	68,9	33,8	5,7	2	0,2	46	3890
Eco Fogo	Capim Branquiaria/Signalgrass	19	16-116	8,3	69,6	7,9	14,2	83,1	16,9	36,4	6	0,4	0,1	48,8	3891
Eco Fogo	Wooden bars, Pinus	20,2	16-117	0,4	80,6	7,4	11,6	87,4	12,6	41,1	6,6	0,9	0,1	50,9	4476
Peat															
Águas Claras	Peat, grain size 0.5 - 2.0 cm	12	16-054	22,6	44,1	1,3	32	58,0	42,0	46,7	4,4	1,2	0,6	24,5	4079
Águas Claras	Peat, grain size 0.5 -4.75 mm	13	16-055	19,1	45	2,4	33,5	57,3	42,7	49,0	4,6	1,2	0,7	25,4	4317
Biomass and Peat-Solid Residues - Results of proximate and elemental analyses and calorific value determination															
Company	Biomass		Sample	Ash	VM	Moist.	FC	VM (%)	FC (%)	C	H	N	S	O	CV(cal/g)
				(%)	(%)	(%)	(%)	d.a.f.	d.a.f.						
Eco Fogo	Sugar Cane Bagasse	8	16-045 -R	31,9	15,4	0,8	51,9	22,9	77,1	58,6	2,3	0,3	0,2	6,7	5079
Eco Fogo	Rice Husks	9	16-046 -R	52,2	30,4	0,7	16,7	64,5	35,5	40,0	1,9	0,3	0,1	5,5	3832
Eco Fogo	Saw Dust	10	16-047 -R	17,2	15	1,4	66,4	18,4	81,6	71,0	2,7	0,1	0,2	8,9	6264
Eco Fogo	wood chips	11	16-048 -R	16,1	15,1	0,2	68,6	18,0	82,0	74,1	2,6	0,2	0,1	6,9	6541
Eco Fogo	Eucalyptus Bark	14	16-111-R	12,2	26,3	3,1	58,4	31,1	68,9	70,5	2,4	0,3	0,1	14,5	5950
Eco Fogo	Eucalyptus Leaves	15	16-112-R	12,2	14,0	1,6	72,2	16,2	83,8	73,3	2,4	1	0,2	10,9	6844
Eco Fogo	Mamona/Castor Bean/Ricinus Communis L.	17	16-114-R	30,8	19,1	2,8	47,3	28,8	71,2	54	2	2	0,5	10,7	5166
Eco Fogo	Capim Camarão/Lollipop Plant	18	16-115-R	29,8	16,9	0,3	53,0	24,1	75,9	53,7	2	1,1	0,3	13,1	4996
Eco Fogo	Capim Branquiaria/Signalgrass	19	16-116-R	23,5	13,7	1,7	61,1	18,3	81,7	53,8	2,6	0,6	0,4	19,1	5763
Eco Fogo	Wooden bars, Pinus	20	16-117-R	1,6	11,8	2,3	84,3	12,3	87,7	74,1	3,1	0,3	0,2	20,7	8031
Peat															
Águas Claras	Peat, grain size 0.5 - 2.0 cm	12	16-054 -R	34,2	45,3	0,1	20,4	68,9	31,1	57,9	2,2	1,5	0,6	3,6	5216
Águas Claras	Peat, grain size 0.5 - 4.75 mm	13	16-055 -R	35,8	47,9	0,3	16,0	75,0	25,0	55,0	2,0	1,3	0,4	5,5	4915

n/a = not analysed

4.3 CHARACTERIZATION OF PEAT FEEDSTOCK AND SOLID RESIDUES (CHARS)

The feedstock peat samples exhibit ash yields of 19.1- 22.6 wt.% (Table 2) and a range of calorific values between 4079 and 4317 cal/g, whereas the solid residues (chars) ash yields vary between 34.2 and 35.8 wt.% (Table 2) and gross calorific values show an increase to 4915 and 5216 cal/g.

4.4 REACTIVITY TO CO₂

The reactivity to CO₂ of feedstock and solid residue (chars) is shown in Table 3. Both feedstock and pyrolyzed coals (solid residues) were the least reactive materials with reactivities ranging from 0.017 to 0.103 min⁻¹ for the former and between 0.011 and 0.086 min⁻¹ for the latter. The results of reactivity to CO₂ of the feedstock coals were inversely proportional to their vitrinite reflectance, which means that coals of higher reflectance showed lower reactivity. Additionally, the reactivity to CO₂ practically did not change before and after vacuum pyrolysis.

Table 3. Reactivity to CO₂ (min⁻¹) of feedstock and pyrolyzed samples.

	Feedstock	Solid residue
Cambui	0.081	0.068
Cruz de Malta	0.017	0.016
Fontanella	0.022	0.019
Mina 101	0.015	0.011
São Vicente do Norte	0.061	0.059
Candiota	0.103	0.086
Peat, grainsize 0.5 - 2.0 cm	0.140	0.119
Peat, grainsize 0.5 - 4.75 mm	0.140	0.107
Sugar Cane Bagasse	0.254	0.096
Rice Husks	0.133	0.048
SawDust	0.267	0.058
Wood chips	0.533	0.079
Eucalyptus Bark	0.767	0.575
Eucalyptus Leaves	0.848	0.594
Signalgrass	0.238	0.152

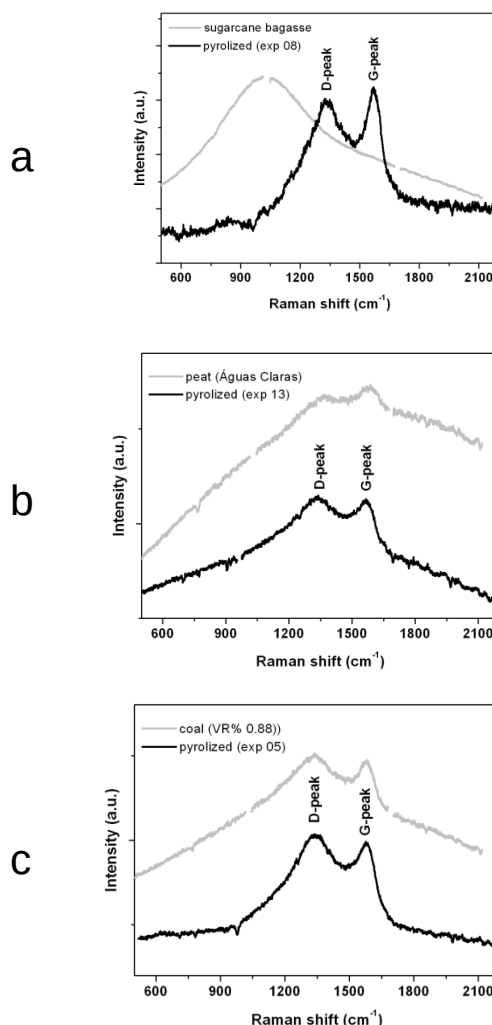
Peat samples had intermediate reactivity values compared to coals and biomasses showing 0.140 min⁻¹ for the feedstock specimens and 0.107-0.119 min⁻¹ for the pyrolysed one. The feedstock biomasses were the most reactive materials with

Eucalyptus leaves being the most reactive sample (0.848 min⁻¹) and rice husks the least reactive biomass (0.133 min⁻¹). The CO₂ reactivity of biomasses substantially decreased in the solid residues compared to the original feedstock, but still remained quite high for some samples, such as Eucalyptus bark and leaves (Table 3).

5 RAMAN SPECTROSCOPY

The samples were measured before and after vacuum pyrolysis. Representative micro Raman spectra of biomass (sugar cane bagasse), peat (Águas Claras) and coal (Seam Barro Branco, Mine 101, Rio Deserto) are shown in Figure 4.

Figure 4: Raman spectra of samples before (grey line) and after (black line) pyrolysis: a) biomass (sugar cane bagasse); b) peat; c) coal (VR% 0.88)



The spectrum of the sugarcane biomass feedstock (Figure 4a) shows only strong fluorescence, with an intensity approximately a

thousand times stronger than the Raman signal of the pyrolyzed material. After pyrolysis distinct Raman peaks can be discriminated, that are related to in plane vibrations of the graphite sheets (G-peak) and to the disorder in the structure when compared to crystalline graphite (D-peak). The peat feedstock shows slight Raman bands over an intense background (Figure 4b) whereas after pyrolysis the G- and D-peaks are much more prominent. The coal feedstock and its pyrolyzed residue (Figure 4c) show almost no changes in the G- and D-peaks, indicating little change in the carbon structure. However the reduction of background indicates loss in hydrocarbons, which present high fluorescence and therefore high background.

6 CONVERSION

6.1 GAS VOLUME GENERATED

The gas volume generated during the pyrolysis experiment was estimated from mass balance (Table 4), subtracting the sum of solid residue (char) and trapped organic matter and water mass from the mass of feedstock used in the experiment.

6.2 CONVERSION TO LIQUIDS

The preliminary results of this study suggest (Table 4) that among the coal samples investigated major conversion to liquids and gases (29.7-33.2 %) occurs in the high volatile bituminous coals from Santa Catarina, whereas in the biomass samples Mamona (Castor Beans), wood chips (Eucalyptus), wooden bars (pinus) and signal grass have all conversion rates > 60 % (Table 4). The conversion rates for the peat samples varied between 32.5 and 46.6 % (Table 4).

7 CONCLUSIONS

7.1 COALS – FEEDSTOCK AND SOLID RESIDUES (CHARS)

Based on vitrinite reflectances a rank range from subbituminous to high volatile bituminous A was established for the coals, except for one sample, showing secondary thermal alteration caused by contact to a volcanic intrusion. The ash yields range from 11.9 to 46.9 wt.%, with calorific values ranging from 2873 to 6484 cal/g. Petrographically the coals are characterized by the predominance of vitrinite macerals. The solid residues show a relative increase in ash yields when compared to feedstock and calorific values between 3076 and 6685 cal/g.

7.2 BIOMASS – FEEDSTOCK AND SOLID RESIDUES (CHARS)

The biomass samples show in general ash yields < 10 wt.%, except for the rice husk and Lollipop plant samples, with calorific values between 3155 cal/g (rice husk) and 4688 cal/g (eucalyptus leaves). The respective solid residues show a relative increase in ash yields, along with a significant increase in calorific values 3832 to 8031 cal/g.

7.3 PEAT – FEEDSTOCK AND SOLID RESIDUES (CHAR)

The peat samples show ash yields between 19.1 and 22.6 wt.%, with calorific values between 4079 and 4317 cal/g. Their respective solid residues are characterized by an increase in ash (34.2 and 35.8 wt%, respectively) and calorific values between 4915 and 5216 cal/g.

7.4 REACTIVITY TO CO₂

Reactivity studies indicate that the reactivity to CO₂ was practically unchanged in the feed coals and their respective solid residues. In the coals the level of reactivity to CO₂ was found to be inversely proportional to their vitrinite reflectance.

In contrast, the pyrolyzed biomass samples (solid residues) showed significant lower reactivity compared to the initial samples. Within the biomass types analyzed Eucalyptus leaves were found to be the most reactive material (0.848 min⁻¹), whereas rice husks were determined as the least reactive biomass (0.133 min⁻¹)

7.5 RAMAN SPECTROSCOPY

The Raman results are consistent with the data obtained from chemical analyses and show a trend of higher graphitization of the carbon structures after vacuum pyrolysis.

7.6 CONVERSION

Conversion rates for gaseous and liquid products were found to be highest in the high volatile bituminous coals from Santa Catarina (29.7 – 33.2 %), whereas highest conversion rates (> 60 %) for the biomass samples were determined for Mamona, wood chips (Eucalyptus), wooden bars (Pinus) and signal grass. The conversion rates for the peat samples varied between 32.5 and 46.6 %.

[illegible]

8 ACKNOWLEDGEMENTS

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