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Articles

Evaluation of atmospheric emissions and ash generated from co-firing of mineral coal with forest residues

Avaliação de emissões atmosféricas e cinzas geradas na cocombustão de carvão mineral com resíduos florestais

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ABSTRACT

The research on alternative energy sources has been motivated by the environmental impacts of non-renewable fuels, which involve polluting gases such as CO_2, CO, SO_2, and NOx. One of the main means of generating sustainable energy using solid fuels is the co-firing of organic waste (biomass) with coal, combined with fluidized bed technology. The aim of this study was to evaluate emissions resulting from the co-combustion of coal and residual organic materials in a 0.25 MWt pilot plant, which consisted of a bubbling fluidized bed reactor (BFBR). In this work, two types of residual biomass from southern Brazil, black wattle bark residue and eucalyptus wood chips, were used.The coal utilized in this study has high levels of sulfur and was sourced from the Candiota mine (CC). By employing a mixture of 75 % RCA and 25 % CC in the reactor feed, gaseous emissions were produced with SO_2 concentrations below the limit established by the environmental legislation (400 mg/Nm^3), resulting in a 90 % reduction compared to the emissions generated from the combustion of pure CC. Tests of co-combustion of biomass with coal demonstrated that concentrations of NOx and CO remained below the emission limit allowed by environmental legislation. The ashes generated during co-combustion processes had high melting temperature values (above 1280 °C), which reduces the risk of problems with encrustation and equipment clogging.

Keywords: Co-firing; Biomass; Fluidized bed; Atmospheric emissions; Ash characterization

RESUMO

A geração de energia a partir de combustíveis não renováveis vem motivando pesquisas que buscam alternativas para a redução de impactos ambientais que envolvem gases poluentes como CO_2, CO, SO_2 e NO_x. Nesse contexto, o uso de resíduos orgânicos (biomassas) na cocombustão com carvão mineral, aliado à tecnologia de leito fluidizado, vem sendo mencionado como um dos principais meios de geração de energia sustentável utilizando combustíveis sólidos. Dessa forma, o objetivo do trabalho foi estudar a cocombustão do carvão mineral e materiais orgânicos residuais em uma planta piloto de 0,25 MW_t, constituída por um reator de leito fluidizado borbulhante (RLFB) para avaliar as emissões dos gases de combustão. Objetivou-se, também, caracterizar os materiais utilizados e as cinzas geradas no processo de cocombustão, visando avaliar possíveis usos das mesmas. No presente trabalho, foram utilizados dois tipos de biomassas residuais da região sul do Brasil: resíduo de casca da acácia-negra (RCA) e cavaco de eucalipto (RCE). O carvão mineral, utilizado neste estudo, contém altos teores de enxofre e foi proveniente da mina de Candiota (CC), localizada no estado do Rio Grande do Sul. Utilizando a mistura de 75 % RCA e 25 % CC na alimentação do reator, obtiveram-se emissões gasosas com concentrações de SO_2 abaixo do limite estabelecido pela legislação ambiental do Rio Grande do Sul (400 mg/Nm^3), significando 90 % de redução em comparação às emissões geradas com a combustão de CC puro. Para todos os testes de cocombustão de biomassas com carvão mineral, as concentrações de NO_x e CO obtidas foram abaixo do limite de emissão permitido pela legislação ambiental. As cinzas geradas nos processos de cocombustão apresentaram altos valores de temperatura de fusão (superiores a 1280 ºC) reduzindo o risco de ocorrência de problemas de incrustação e entupimento dos equipamentos.

Palavras-chave: Cocombustão; Biomassa; Leito fluidizado; Emissões atmosféricas; Caracterização das cinzas

1 INTRODUCTION

The use of fossil fuels for energy generation and its impact on the environment have prompted research into mitigating atmospheric emissions and promoting sustainable use of non-renewable energy sources. Coal is the most abundant and long-lasting non-renewable energy resource and is one of the world's most significant energy reserves in the long term. Coal provides one-third of the world's energy and accounts for 38.5 % of electricity generation. It also plays a crucial role in sectors such as the iron and steel industry (International Energy Agency, 2021).

Fluidized bed technology provides significant advantages in energy conversion compared to traditional methods like fixed beds. This approach offers flexibility in fuel usage, enabling efficient combustion of low-quality solid materials, such as coal with high ash content and biomass with high moisture content. These benefits arise from the uniformity in operating temperature, as noted by Flegkas *et al*. (2018) and Wander *et al*. (2020). The underlying principle involves the flow of fluid through a bed of particles, where they become suspended when the fluid reaches a velocity sufficient to overcome the pressure drop of the medium. This phenomenon is known in engineering as the fluidization state.

Researchers have been studying the use of different types of biomass in co-firing with coal in fluidized bed systems. The main parameters analyzed in these studies are: combustion characteristics (Goldfarb; Ceylan, 2015) and the ash formed (Henne *et al*., 2020), fluid dynamics, bed segregation (Kuprianov; Janvijitsakul; Permchart, 2006), atmospheric emissions (Nunes; Matias; Catalão, 2014; Kumar; Mohapatra; Singh, 2015) and raw material availability (Dumortier, 2013). In general, fluidized bed technology is considered one of the primary methods for generating sustainable energy through the combustion of solid fuels. This is due to its flexibility, stability, and efficiency (Khan *et al*., 2009).

As an ecological energy source, biomass consists mainly of organic materials with a polymeric composition, such as cellulose, hemicellulose and lignin, which vary in relative amounts for each type of compound. One of the advantages of using biomass in combustion processes is the possibility of neutralizing greenhouse gas emissions such as CO_2. This is due to the fact that there is a zero balance of CO_2 emissions when biomass is burned, as there is a direct relationship between the amount of carbon dioxide released into the atmosphere during combustion and the amount absorbed from the atmosphere by the vegetation (biomass) through photosynthesis. Another advantage of using biomass is the ability to use agricultural, forestry, industrial and municipal waste that has a biodegradable fraction that can be used as fuel (Eom; Kim; Lee, 2011; Jiang *et al*., 2013). The use of biomass to burn mineral coal with a high sulfur content can minimize emissions of pollutants such as sulfur dioxide (SO_2). This is due to the low sulfur content found in biomass, unlike the high sulfur content found in coal from Brazilian reserves.

In recent years, biomass has become a prominent energy source. Brazil generates significant annual volumes of organic waste with energy potential, such as sugarcane bagasse, rice husks, black wattle bark, and eucalyptus chips. Rio Grande do Sul is known for its annual production of 250,000 tons of black wattle bark waste (Acacia mearnsii De Wild), which is derived from the tannin extraction industry. Additionally, the state leads the country in cultivating this legume, with forests of the species occupying 75,000 hectares. These forests are of growing global interest for their cellulosic pulp, which is used in the production of cellulose via the kraft process under various cooking conditions (Giesbrecht *et al*., 2022). It is known that this biomass is lignocellulosic and is mainly structured by three polymers: cellulose, hemicellulose and lignin. In addition, other compounds such as acetyl groups and minerals are present in small amounts and contribute to a complex architecture.

In the southern region of Brazil, natural and geographical conditions favor the cultivation of eucalyptus (Eucalyptus spp.), also a type of lignocellulosic biomass that is important in the Brazilian scenario, both in terms of cultivation and energy use. Eucalyptus is well-suited to the local climate and soil. It has a calorific value ranging from 16.46 MJ/kg to 19.17 MJ/kg (Neiva; Furtado; Finzer, 2018). In comparison, coal from the Candiota mine, which has a high ash content, has a calorific value ranging from 10.26 MJ/kg to 14.46 MJ/kg (Kalkreuth *et al*., 2013; Faé Gomoes, 2014). Although eucalyptus cultivation only covers approximately 1% of the national territory, it accounts for over 90 % of the total production of wood for industrial purposes (Maggi *et al*., 2018). Therefore, eucalyptus wood waste presents opportunities for energy use, with briquetting and chips being efficient ways of concentrating energy in forest biomass (Souza *et al*., 2022).

Given the aforementioned considerations, it is crucial to conduct research that enables the utilization of high-sulfur mineral coal and forest residues in pilot plant systems as potential alternatives for energy generation. The objective of this study was to evaluate flue gas emissions resulting from the co-combustion of mineral coal with residual organic materials, specifically black wattle bark and eucalyptus chips, in a 0.25 MWt pilot plant using a bubbling fluidized bed reactor (BFBR). The objective of this study was to characterize the materials used and the ash generated during the co-firing process to evaluate its potential uses.

2 MATERIALS AND METHODS

This study utilized two types of biomasses that are commonly found in southern Brazil: black wattle bark residue (RCA) from the tannin extraction industry and eucalyptus chips (RCE) from the pulp and paper industry. The coal used in this study was obtained from the Candiota mine (CC), which is the largest coal deposit in Brazil and is located in the southern region of the state of Rio Grande do Sul. The experiment was conducted at the Process Engineering Department of CIENTEC, which was formerly a Science and Technology Foundation in the state of Rio Grande do Sul. The advanced campus is located in the municipality of Cachoeirinha.

2.1 Raw material characterization

The fuels CC, RCA and RCE underwent immediate analysis, elemental analysis, and lower heating value (LHV) characterization. Samples were taken from a stored batch of approximately 100 kg, from which 10 portions were randomly collected at different points. The fractions from each batch were combined, homogenized, and divided into quarters. A single sample was created for analysis and characterization. All fuel characterization tests were conducted following ASTM D7582-15, D5373-08, D4239-11, and ABNT-NBR 8633-84 standards. Ash fusibility tests were performed in accordance with ASTM D1857/D1857M-18. The pilot plant tests were conducted only once for each operating condition due to the time required to reach the reactor’s operating temperature of approximately 850 ºC. Additionally, the fibrous characteristics of the biomass caused problems with the reactor’s feed screw locking, making it challenging to conduct duplicate tests.

Thermogravimetric tests (TGA) were conducted to evaluate the thermal degradation of the raw materials. The tests were performed using a thermobalance (Perkin Elmer, model TGA7) in a synthetic air atmosphere, ranging from room temperature to 900 °C. Each sample, weighing 15 mg, was placed in a support crucible. The flow of synthetic air was maintained at 20 mL/min. The sample was heated from room temperature to 200°C at a rate of 5°C/min. Once the temperature stabilized at 200°C, the heating rate was increased to 10°C/min until the temperature reached 900°C.

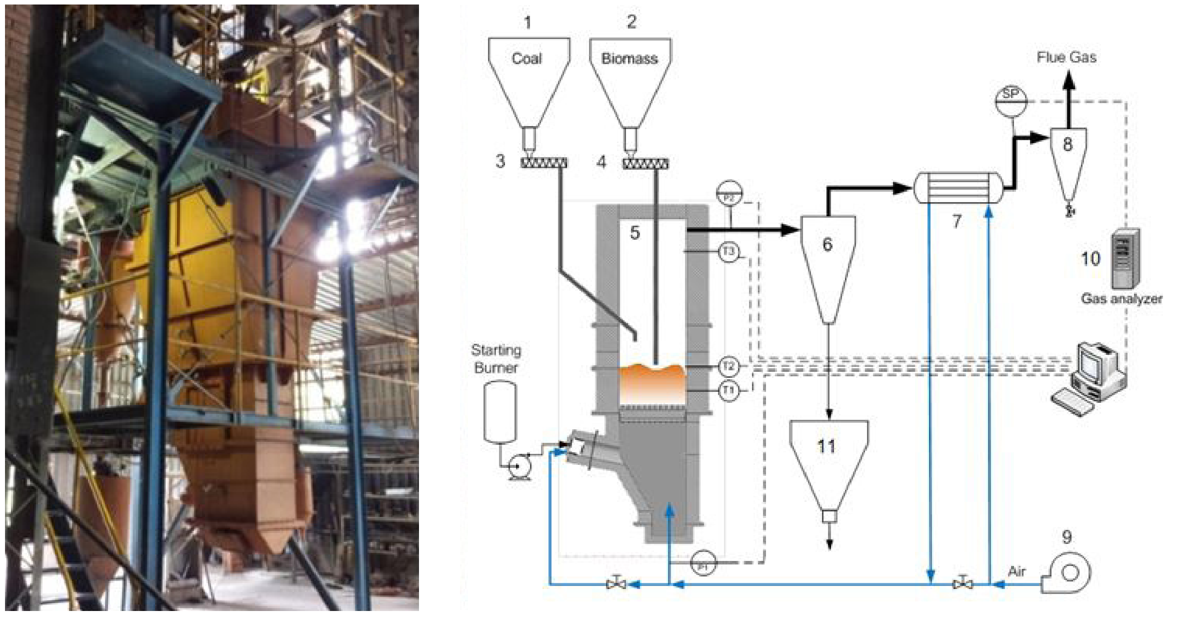
Fly ash and fluidized bed ash generated during the experimental process at the pilot plant were collected and analyzed using X-ray fluorescence (XRF) and X-ray diffraction (XRD). The fusibility characteristics of the ashes were assessed using a melting temperature analyzer microscope due to the potential presence of melting oxides in biomass ash. The temperatures for deformation (DT), ball (ST), semi-sphere (HT), and melting (FT) were obtained by heating at a rate of 10 K/min up to 1500ºC.

2.2 Pilot plant tests with fluidized bed reactor

The co-firing tests were conducted in a 0.25 MW_t pilot plant that was equipped with a bubbling fluidized bed reactor (BFBR) system. Figure 1a displays the pilot plant reactor, which has an external height of 2.8 m and a carbon steel combustion chamber lined internally with refractory material. The bed's interior measures 40 cm in height, 27 cm in width, and 97 cm in length. It is filled with sand that has an average particle diameter of 0.45 mm. Figure 1b displays the process flowchart, including the primary equipment and accessories.

During the operations, CC coal stored in silo (1) and one type of biomass at a time (RCA or RCE) stored in silo (2) are conveyed at controlled rates via screw conveyors (3 and 4) to the BFBR furnace (5) where co-firing occurs. The gases rise through the reactor and pass through the cyclone (6) to separate the ash. The ash is then deposited in the ash silo (11). The combustion gases are preheated by the heat exchanger (7) using the heat generated by the gases. The preheated air is then supplied to the fluidized bed. The gases are released into the chimney after passing through a new cyclone (8). The air that feeds the system is supplied by the forced draught fan (9). Figure 1 shows the reactor pressure measurement points as P1 and P2. The flue gas sampling point (SP) is located after the heat exchanger (7) and is connected to a gas analyzer (10) (HORIBA CMA-680). Thermocouples T1 and T2 record the temperature inside the bed and in the fluidization zone. The supervisory system was used for supervision, control, and data acquisition.

Figure 1 – Shows the pilot plant used for the experimental tests. Panel (a) displays a photo of the BFBR, while panel (b) shows the flowchart of the co-firing process with the system's main equipment



(a) (b)

Source: Authors (2018, 2023)

To replicate standard operating conditions, we conducted operations in the pilot plant using only CC coal. We conducted co-firing tests by adjusting the feed mass ratios between the biomass (RCA and RCE) and the coal (CC). The bed was continuously fed with compositions containing 40% and 60% eucalyptus residue by mass in relation to the coal. These samples were labeled RCE40 and RCE60, respectively. Additionally, a firing condition using 100 % eucalyptus residue in the feed was tested and labeled as sample RCE.

Tests were conducted using charcoal (CC) and black wattle bark residue (RCA) in proportions of 40%, 60%, and 75% by mass of RCA in relation to charcoal. The samples were named RCA40, RCA60, and RCA75, respectively. It was not possible to carry out a comparison operation using 100% RCA in the reactor due to problems with clogging and jamming of the feed screw caused by the fibrous nature of the material. The operating temperature of the system was determined and data was collected after the temperature in the bed (T1 and T2) had stabilized at approximately 850ºC. It is important to note that the optimal temperature for complete combustion of solid fuels in BFBR beds is around 850 ºC (Gomes, Vilela, & Zen Osório, 2013). This temperature promotes complete calcination and favors the thermodynamic conditions of the sulfation reaction, resulting in a low concentration of SO_2 being released into the exhaust gas (VALK, 1995). The feeding conditions in the BFBR lasted between 60 and 80 minutes after the operating conditions stabilized.

Data collection was conducted after stabilizing the bed's operating conditions. The mass flow rates of the fuels (coal and biomass) were set, and the reactor's air flow rate was adjusted between 191.6 Kg/h and 243.0 Kg/h to maintain the temperature inside the fluidized bed close to 850°C.

The mass proportions of eucalyptus chips (RCE) in relation to mineral coal (CC) were 40%, 60%, and 100 %. The tests with black wattle bark residue (RCA) were carried out using mass proportions of 40%, 60%, and 75% of RCA in relation to CC. The operation using 100% RCA as feed for the reactor was not possible due to clogging and jamming of the reactor's feed screw caused by the fibrous nature of the material. To compare co-firing results, a benchmark test was conducted using 100 % CC coal The combustion efficiencies (η) at the different operating conditions were calculated according to Despina Vamvuka *et al*. (2012), as shown in Equation (1):

$$\eta(\%) = 100 - \frac{CO_2}{CO_2 - CO} - L_{cv} - L_{cf} \tag{1}$$

Where: L_{cv} represents the unburned carbon in the fly ash, determined experimentally by TGA analysis using the fly ash collected in the ash silo (11) after the separation cyclone (6) in Figure 1b; The term L_{cf} represents the unburned carbon of the heavy ash that settles inside the bed.

After each test, weighed ash samples were collected and analyzed for carbon content. As expected for this type of combustor, the carbon contents for bottom ash were low for all samples (less than 0.5 % by weight). The terms CO and CO_2 represent the contents of these compounds that are present in the emissions generated, obtained using the gas analyzer.

3 RESULTS AND DISCUSSIONS

Regarding particle size, the average diameter of Candiota coal (CC) was 0.35 mm. The evaluated biomasses, eucalyptus chip residue (ECR) and black wattle bark residue (WBA), had irregular shapes but sizes ranging from 1 to 5 mm in thickness and 5 to 10 mm in width.

Table 1 shows the results of the immediate analysis, elemental analysis and lower calorific value (LHV) of the fuels used in the research.

Table 1 – Immediate analysis, elemental analysis and lower heating value (LHV) of forest residues RCA, RCE and charcoal CC

Analyses	**RCA**	**RCE**	**CC**
Immediate (%)			
Fixed Caron [a]	20.0	12.2	21.9
Volatile material [a]	76.2	82.5	23.1
Ashes [a]	3.8	5.3	55.0
Humidity	12.0	14.6	17.1
Elementary [a] (%)			
C	51.3	47.8	33.8
H	4.1	5.4	2.3
S	0.1	0.1	1.9
N	1.4	0.1	0.6
O	39.4	41.4	61.4
Lower heating valu (kJ/kg)			
PCI [a]	18430	17115	12205

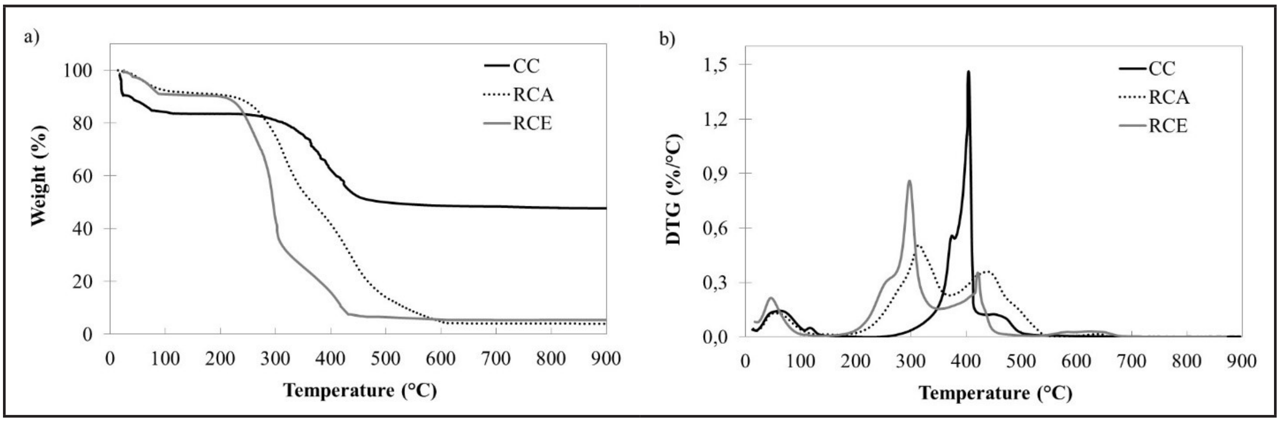
Source: Authors (2023)

In where: [a] dry base

Table 1 shows that mineral coal has a high ash content of 55 % by mass and a significant sulfur content of 1.9 %. In contrast, biomass has a higher volatile matter content and a higher carbon content, resulting in a higher calorific value than coal. The fuel characterization results are consistent with previous studies conducted by Faé Gomes (2013) who used black wattle bark and Candiota coal to investigate cleaner production using coal and biomass, and with the study by Nunes (2016) who also used Candiota coal to evaluate the combustion kinetics of coals with high ash content.

The mass loss of the RCA, RCE, and CC fuel samples was observed as a function of temperature increase through thermogravimetric (TGA) and derived thermogravimetric (DTG) analyses, as depicted in Figure 2. The combustion process of lignocellulosic biomass can be divided into four main zones: removal of moisture and very light volatile components (<120°C); degradation of hemicellulose (220 – 315 °C); decomposition of cellulose and lignin (~315 – 400°C) and degradation of lignin (> 400°C) (Sanchez-Silva, 2012; Asadieraghi; Wan Daud, 2014). This last zone refers to the stabilization of the mass associated with unconverted solid carbon (Crelling, 1992; Silva; Milioli, 2008; Ávila, 2017).

Figure 2 – Thermogravimetric mass loss curves (a) TGA and (b) DTG of Candiota coal (CC), black wattle bark residue (RCA) and eucalyptus chip residue (RCE)



Source: Authors (2023)

Figure 2a shows that RCA and RCE biomass experience mass losses of approximately 10 % up to 100°C due to humidity. Between 250°C and 600°C, there is another mass loss related to the decomposition of cellulose, hemicellulose, and lignin. After reaching 450°C and 600°C, the mass loss stabilizes for RCE and RCA, respectively, which is attributed to the ash content of the materials (Pécora *et al*., 2014). Figure 2a shows the analysis of CC, which exhibits a less pronounced mass loss compared to RCE and RCA biomasses. However, three mass loss events can be observed for CC charcoal: loss of moisture up to 110°C (around 20 % mass loss), oxidation of hydrocarbons and volatile matter between 300°C and 450°C (45 % mass loss), and stabilization of the mass related to charcoal ash after this point (Ávila *et al*., 2017).

Figure 2b shows that the highest degradation rates of RCE and RCA biomass correspond to hemicellulose degradation at a temperature close to 300 °C. The mass loss derivative graph in Figure 2b does not show a clear peak for cellulose degradation in RCA and RCE. This may be due to cellulose degradation occurring simultaneously with lignin degradation, resulting in overlapping peaks in this degradation range (Huang *et al*., 2011). Furthermore, the devolatization of RCA lignin exhibited a slower rate of decomposition, as indicated by the broader 'shoulder' in Figure 2b, spanning temperatures from 450°C to 550°C. Lignin is a reinforcing component of the cell wall and has a more stable thermally complex structure than hemicellulose and cellulose. Therefore, its complete degradation occurs at higher temperatures (Vamvuka *et al*., 2003; Silva *et al*., 2003; Pinheiro *et al*., 2003; Nogueira *et al*., 2003; Pinho *et al*., 2003; Van *et al*., 2010; Baeyens *et al*., 2010; Brems *et al*., 2010; Janssens *et al*., 2010; Dewil *et al*., 2010; Teixeira, 2015). The temperature values above 430°C for RCE and 550 °C for RCA indicate the stabilization of the mass associated with the inorganic material (ash) content of the materials (Edreis, 2013; Pécora *et al*., 2014). The TGA and DTG analysis data for the biomasses support those determined by the immediate analysis shown in Table 1.

3.1 Co-combustion tests and generated emissions

Table 2 displays the experimental matrix and operating data for the tests conducted in the bubbling fluidized bed reactor (BFBR). The fuel feed rates, expressed in Kg/h, correspond to the ratio of 'Mass of Biomass per Mass of Mineral Coal' represented by the term '% m/m'. The table also includes the average temperatures inside the bed obtained during each test in the BFBR.

Table 2 – Experimental matrix used in this study

Fuel	**Test name**	**Biomass/ coal feed (% m/m)**	**Coal feed rate (kg/h)**	**Biomass feed rate (kg/h)**	**Air flow (kg/h)**	**Bed temperature (°C)**	
						T1	T2
CC	CC	0/100	33.10	-	223.9	847	855
CC and RCA	RCA40	40/60	15.90	10.1	191.6	847	853
CC and RCA	RCA60	60/40	10.70	16.2	199.0	850	856
CC and RCA	RCA75	75/25	6.00	23.4	195.6	853	859
CC and RCE	RCE40	40/60	16.47	10.8	221.3	851	859
CC and RCE	RCE60	60/40	11.00	17.5	216.9	847	855
RCE	RCE	100/0	-	28.2	243.0	854	858

Source: Authors (2023)

Table 3 displays the average concentrations of SO_2, NOx, and CO gases, on a dry basis, produced during the co-firing process and combustion with CC. The concentration of SO_2 decreases significantly as the fraction of both biomasses in the reactor feed stream increases. During the RCE40 and RCE60 tests, where 40 % and 60 % eucalyptus residue was used, the SO_2 concentrations exceeded the emission limit (400 mg/Nm^3) set by the Rio Grande do Sul environmental agency. However, in the RCA60 and RCA75 tests, where 60 % and 75 % acacia bark waste was used, the SO_2 concentrations remained below the permitted limit. Furthermore, in comparison to the emissions produced by burning pure CC fuel, the addition of acacia bark at the specified concentrations resulted in a noteworthy 93 % reduction in emissions of this compound. These findings suggest that the inclusion of acacia bark can significantly decrease the SO_2 emissions generated while maintaining operational stability. However, additional tests utilizing RCA and RCE biomass are necessary to investigate the synergy between them and co-firing with CC coal.

The lower concentration of SO_2 in the gases produced can be explained by the fact that the biomasses (RCA and RCE) have a low sulphur content of 0.1 %, compared to the 1.9 % content of mineral coal (CC) shown in Table 1. Other studies, such as that by Atimtay and Kaynak (2008) using peach pit residues and that by Varol et al. (2018) using wooden pallets, have shown similar results in SO_2 concentrations in emissions generated during the co-firing process with mineral coal.

Table 3 – Concentration of SO_2, NOx and CO in the flue gases of CC and in the tests with RCE and RCA in the feed and combustion efficiency η_c

Fuels	Test name	Biomass/ Coal feed (% m/m)	SO_2 (mg/Nm³)	NO_x (mg/Nm³)	CO (mg/Nm³)	Combustion Efficiency η_c (%)
CC	CC	0/100	3520	249	139	93.0
CC e RCA	RCA40	40/60	506	320	159	92.8
CC e RCA	RCA60	60/40	297	333	164	93.0
CC e RCA	RCA75	75/25	246	349	151	93.0
CC e RCE	RCE40	40/60	2595	329	101	95.4
CC e RCE	RCE60	60/40	1153	318	71	95.7
RCE	RCE	100/0	292	211	58	95.8

Source: Authors (2023)

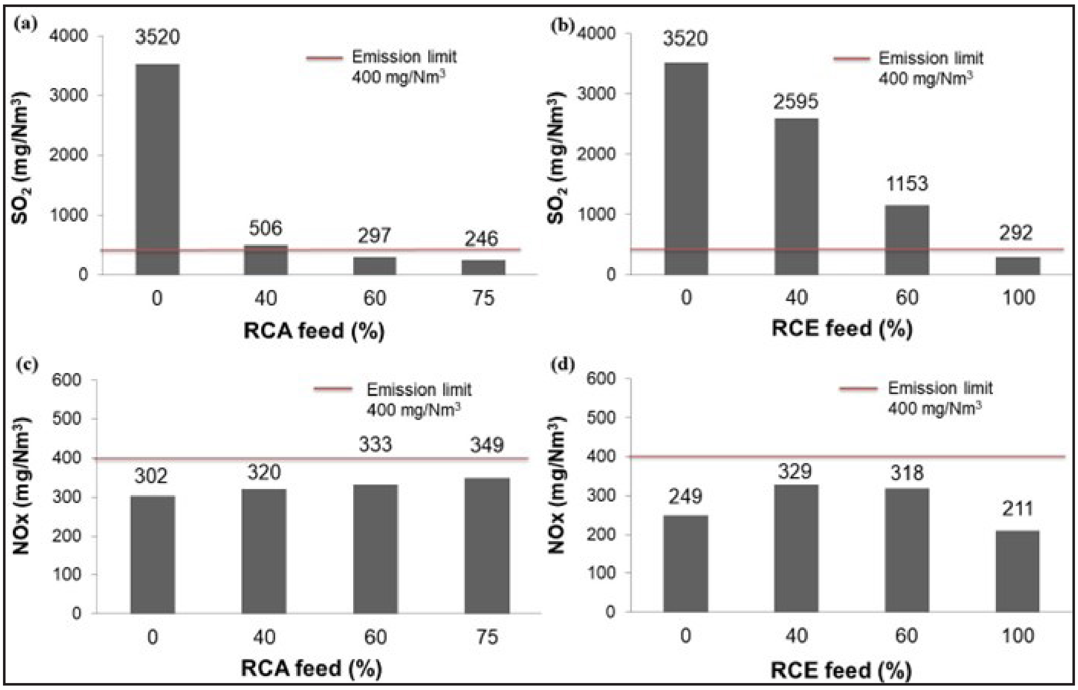
In where: Emission limits established by environmental legislation: SO_2 = 400 mg/Nm³, NOx = 400 mg/Nm³ and CO= 250 mg/Nm³ (FEPAM, 2018).

The experiments conducted in the reactor with varying proportions of RCE and RCA did not result in significant changes in NOx emissions (refer to Table 3). This is due to the relatively low bed temperatures at which the experiments were conducted, approximately 847 to 859 °C, which do not favor the formation of thermal NOx. Instead, nitrogen from the fuel itself is utilized (Van Loo; Koppejaan, 2008; Liu *et al*., 2013). Thus, at the highest fuel concentrations, NOx emissions from the co-firing of RCA were higher than those from RCE due to the higher nitrogen content present in the former, as shown in Table 1. However, all resulting NOx concentrations remained below the regulatory body's emission limit of 400 mg/Nm³ (FEPAM, 2018). Thus, at the highest fuel concentrations, NOx emissions from the co-firing of RCA were higher than those from RCE due to the higher nitrogen content present in the former, as shown in Table 1. However, all resulting NOx concentrations remained below the regulatory body's emission limit of 400 mg/Nm³ (Madanayake *et al*., 2017).

The CO concentration values in the emissions, as a function of the biomass feed rate in the reactor, were between 164 and 58 mg/Nm³ for the RCA60 and RCE co-firing tests (Table 3). These concentrations can be considered low when analyzing the properties of the biomasses and comparing them, for example, to the values obtained by Sun *et al*. (2013). The authors of the original study utilized sub-bituminous coal and sawdust as fuels in a circulating fluidized bed reactor, which have similar characteristics to those used in this study. The results obtained by the authors showed a range of 220 to 290 ppm (250 to 320 mg/m^3) with a feed rate of up to 25% biomass in the reactor. However, the BFBR system can operate under optimum excess air conditions without altering the temperature in the fluidized bed. This allows for the maintenance of operating standards and high combustion efficiencies, as shown in Table 3. Therefore, co-firing biomass provides a significant reduction in SO_2 emissions without causing high CO.

Figures 3(a), 3(b), 3(c) and 3(d) display the concentrations of SO_2 and NOx for the CC/RCA and CC/RCE mixtures tested in the co-firing experiments. It is evident that, in all experiments, the emission limits for SO_2 and NOx remained below the values established by environmental legislation, except for the SO_2 emissions in the test with RCE40 and RCE60 (40 % and 60 % eucalyptus in the feed, respectively), which exceeded the limit established by FEPAM (2018). Furthermore, the test using 40 % RCA in the feed (RCA40) exhibited emissions slightly exceeding the environmental limit. Additionally, Figures 3c and 3d demonstrate that the NOx concentration values in the emissions remained below the emission limit set by environmental legislation (400 mg/Nm^3) for all test conditions.

Figure 3 – SO_2 and NOx concentrations for the mixtures of CC and RCA and CC and RCE applied in the pilot plant co-firing tests

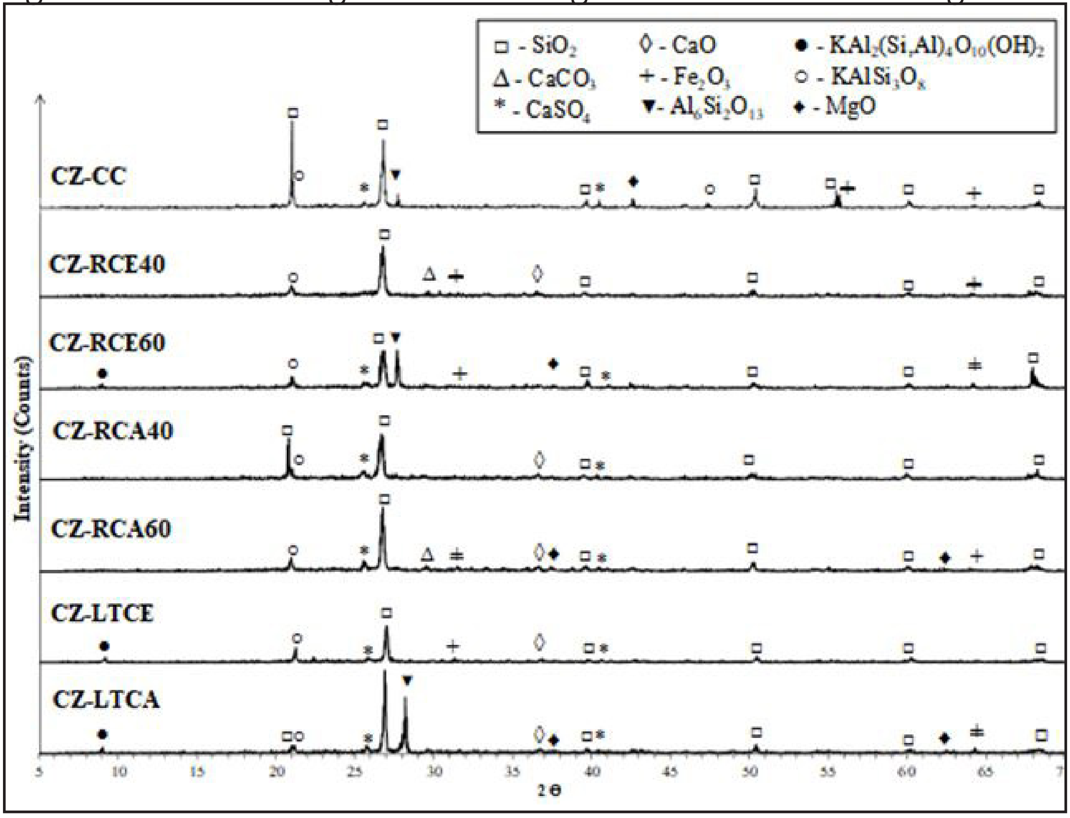


Source: Authors (2023)

3.2 Ash characterization

Figure 4 displays the X-ray diffractograms (XRD) of the CZ-CC sample (fly ash from the CC combustion tests), the CZ-RCE40, CZ-RCE60, CZ-RCA40 and CZ-RCA60 (fly ash from the RCE40, RCE60, RCA40 and RCA60 combustion tests, respectively), and the fluidized bed bottom ash samples after the CC and RCA tests (CZ-LTCA) and after the CC and RCE tests (CZ-LTCE). The diffractograms show that quartz (SiO2) is the primary phase in the ash. The characteristic peaks of SiO_2 decreased when biomass partially substituted coal in the samples for co-firing. In addition to quartz, the ash contains calcium oxide (CaO), aluminum silicate ($Al_6Si_2O_{13}$), alkali feldspar ($KAlSi_3O_8$), and anhydrite ($CaSO_4$). No magnesium sulphates were identified in the diffractograms of the ashes analyzed. This is probably because the sulphates of other elements, such as $MgSO_4$, $Al2(SO_4)_3$, $Fe_2(SO_4)_3$ and K_2SO_4, are thermally less stable when compared to $CaSO_4$ (Cheng *et al*., 2003; Shao *et al*., 2012; Fae *et al*., 2016).

Figure 4 – XRD diffractograms of the ash generated after the co-firing tests in the pilot plant



Source: Authors (2023)

In where: CZ-CC = Candiota coal ash; CZ-RCE40 = 40 % RCE co-firing ash; CZ-RCE60 = 60 % RCE co-firing ash; CZ-RCA40 = 40 % RCA co-firing ash; CZ-RCA60 = 60 % RCA co-firing ash; CZ-LTCE = bed ash after RCE co-firing tests and CZ-LTCA = bed ash after RCA co-firing tests.

The diffractograms of the 60 % RCA biomass ash (CZ-RCA60) and the bed ash showed the presence of a new crystalline phase, calcium oxide (CaO), which was not found in the CC combustion ash (CZ-CC). Additionally, the compound magnesium oxide (MgO) was identified in the fly ash from the tests carried out with higher percentages of biomass (CZ-RCA60 and CZ-RCE60) and in the bed ash after the tests with RCA (CZ-LTCA). Magnesium oxide is a secondary mineral that may have formed from the decomposition of dolomite or from the oxidation of magnesium present in the biomass composition (Varol; Atimtay; Olgun, 2014). Figure 4 shows diffractograms of all samples, except for CZ-RCE40, which did not show peaks of anhydrite ($CaSO_4$). Aluminum silicate in the form of muscovite ($KAl_2(SiAl)_4O_{10}(OH)_2$) was identified in the ash from the 60 % RCE co-firing (CZ-RCE60) and in the bed ash after testing with CC and RCA (CZ-LTCA) and after testing with CC and RCE (CZ-LTCE). The presence of muscovite in the ash is significant because it can be utilized in construction, electronics manufacturing, or even in the synthesis of zeolites.

Table 4 shows the results of the X-ray fluorescence analysis of the fly ash. It can be seen that the ash from burning coal (CC) has the highest levels of silicon oxide (SiO_2) compared to the other samples. The co-firing ashes (CZ-RCA40, CZ-RCA60, CZ-RCE40, and CZ-RCE60) contain high levels of silicon, aluminum, iron, calcium, and magnesium oxides. The increase in the content of iron, calcium, and magnesium oxides is due to the presence of these elements in the biomass composition. In contrast, the content of silicon oxide is lower compared to coal ash (CC). The results of the XRD diffractograms shown in Figure 4 corroborate the effectiveness of the desulphurization process. The analysis did not reveal significant levels of alkaline compounds, such as potassium and sodium, which could lead to the formation of deposits in the gas outlet system. Nunes (2019) found that high levels of sodium oxide were associated with the formation of slag and scale in boilers, but this was not observed in our study.

Table 4 – XRF analysis of the ash samples from the pilot plant tests

Oxides	**Fly ash**					**Bottom Ash (Bed)**	
(%)	**CZ-CC**	**CZ-RCA40**	**CZ-RCA60**	**CZ-RCE40**	**CZ-RCE60**	**CZ-LTCA**	**CZ-LTCE**
SiO_2	66.67	39.73	40.80	49.52	41.18	48.56	46.92
Al_2O_3	17.69	13.85	15.53	28.05	19.50	17.81	21.19
Fe_2O_3	5.24	9.36	9.97	9.69	17.53	7.29	8.48
CaO	2.92	15.94	19.14	4.40	9.03	14.90	12,34
MgO	0.25	2.03	2.22	1.22	1.85	4.47	2.30
SO_3	3.64	14.92	7.75	3.36	3.82	4.70	4,75
K_2O	2.31	1.67	2.00	1.92	3.44	1.78	2.25
TiO_2	1.09	1.46	1.40	1.14	2.58	0	0.97
Na_2O	0	0.24	0.26	0.26	0,53	0.23	0.30
ZrO_2	0.02	0.06	0.06	0.11	0.11	0.08	0.06
P_2O_5	0.07	0.37	0.43	0.07	0.13	0	0.07
ZnO	0.02	0.07	0.12	0.06	0.09	0.04	0.12
MnO	0.04	0.10	0.10	0.05	0.11	0.06	0.11
SrO	0.01	0.17	0.15	0.05	0.06	0.04	0,06
Rb_2O	0.01	0.03	0.03	0.03	0	0.03	0.04
Y_2O_3	0.01	0	0	0.03	0.04	0.01	0.01
NiO	0.00	0	0.03	0.03	0	0	0.03
Co_2O_3	0	0	0	0	0	0	0
CuO	0	0	0	0	0	0	0
Cr_2O_3	0.02	0	0	0	0	0	0

Source: Authors (2023)

In where: CZ-CC = Candiota coal ash; CZ-RCE40 = 40 % RCE co-firing ash; CZ-RCE60 = 60 % RCE co-firing ash; CZ-RCA40 = 40 % RCA co-firing ash; CZ-RCA60 = 60 % RCA co-firing ash; CZ-LTCE = bed ash after RCE co-firing tests and CZ-LTCA = bed ash after RCA co-firing tests.

The analyses of the bed ash samples collected after the co-firing tests with the RCA (CZ-LTCA) and RCE (CZ-LTCE) biomasses are shown in Table 4. Silicon (SiO_2) and aluminum (Al2O$_3$) oxides predominate. With regard to alkaline oxides, relatively low concentrations were found for sodium (Na_2O) and potassium (K_2O) oxides. The utilization of RCA and RCE biomass in the co-firing process with Candiota coal can reduce or prevent issues related to deposit formation. As per Vassilev's research (2013), coarse ash particles, consisting of primary and secondary elements found in biomass, such as Ca, Mg, Si, Al, and some unburned organic material, are retained in the flue gas and deposited in the bed.

Table 5 displays the melting temperatures of the fly ash and bed ash samples. The values for the initial deformation temperature (DT), softening temperature (ST), hemispherical temperature (HT), and flow temperature (FT) were higher than those found by Rizvi et al. (2015), who studied the melting temperatures of ash from wood waste, peanut shells, sunflower shells, and vegetable waste (grasses) used in energy generation. The melting temperatures of inorganic compounds in ash are linked to the presence of certain elements. For example, $KAlSi_2O_6(S_2)$, $Ca_2MgSi_2O_7(S)$, and $Ca_3Fe_2Si_2O_{12}(S)$ are compounds that can cause fusion at lower temperatures, typically between 1100 °C and 1300 °C (Rizvi *et al.*, 2015). However, the ash sampled did not show the formation of the inorganic compounds mentioned, despite the high concentrations of Si, Al, and Fe oxides and the presence of S, Ca, and K oxides (Table 4).

Table 5 shows that the fusion temperature (FT) of the CC ash was above 1500°C, while for the co-firing ash, the FT values ranged from 1284°C to 1457°C. The high FT values of the biomass ash with the Candiota coal are important because they reduce the risk of problems associated with the formation of deposits and agglomeration in the equipment. The fluidized bed co-combustion process takes place at temperatures around 850°C.

Table 5 – Melting temperature of CC ash, CC co-firing ash with RCA and RCE biomass and fluidized bed bottom ash

Test nomenclature	DT (°C)	ST (°C)	HT (°C)	FT (°C)
CZ-CC	1161	1217	1225	>1500
CZ-RCA40	1329	1381	1417	1457
CZ-RCA60	1279	1374	1410	1436
CZ-RCE40	1234	1247	1257	1284
CZ-RCE60	1183	1253	1268	1312
CZ- LTCA	1187	1246	1255	1367
CZ-LTCE	1210	1257	1266	1384

Source: Authors (2023)

In where: DT = Deformation temperature (°C); ST = Sphere temperature (°C); HT = Semisphere temperature (°C); FT = Melting temperature (°C); CZ-CC = Candiota coal ash; CZ-RCE40 = 40 % RCE co-firing ash; CZ-RCE60 = ash from 60 % RCE co-firing; CZ-RCA40 = ash from 40 % RCA co-firing; CZ-RCA60 = ash from 60 % RCA co-firing; CZ-LTCE = bed ash after RCE co-firing tests and CZ-LTCA = bed ash after RCA co-firing tests.

Based on the characterization results, it was found that the ash generated has a high melting temperature of over 1280 ºC. This reduces the risk of equipment fouling and clogging when co-firing RCA and RCE biomass with Candiota coal (CC). Due to its high melting temperature, it is unlikely that any issues related to deposit formation in the equipment will occur during the fluidized bed co-firing process, which takes place at temperatures around 850 °C. Finally, this study recommends that a pilot-scale bubbling fluidized bed reactor can operate within the emission limits established by environmental legislation using the co-firing process of domestic coal with forest biomass residues, such as black wattle bark, which have a high sulphur and ash content. The pilot system can operate with excess air patterns without altering the temperature in the fluidized bed, thereby maintaining stable operating conditions and high combustion efficiencies. This study can serve as a foundation for pilot and industrial scale developments in bubbling fluidized bed applications.

4 CONCLUSIONS

The bubbling fluidized bed reactor in pilot plant scale was successfully tested for the co-firing process of Candiota mineral coal (CC) with high sulfur content, ash, and forest biomass of eucalyptus wood chips (RCE) and black wattle husk (RCA). Diverse mass fractions of coal and biomass were fed into the reactor, and operational parameters of the co-firing process and gas emissions were evaluated, as well as the characterization of fuels and generated ashes.

The co-firing of mineral coal (CC) and acacia bark residue (RCA) resulted in a reduction exceeding 90% in the concentration of SO_2 compound in the produced gases, compared to the use of pure coal. The values obtained for the compound were below the emission limit established by the environmental agency of Rio Grande do Sul.

Regarding the concentration of NOx and CO compounds in the combustion gases, the tests showed no significant variations in emissions, with levels considered low and within the limits established by the environmental agency. Thus, the co-combustion of coal with biomass resulted in a significant reduction in SO_2 emissions without causing a considerable increase in NOx and CO emissions.

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