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DEVELOPMENT OF CO2 SORBENTS DOPED WITH RICE HUSK ASH APPLIED TO CALCIUM LOOPING

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Abstract. *Brazil has a great potential for the production of combustible gases (syngas) from various types of biomass. Therefore, the use of biomass will be encouraged in the coming years, since CH_4 is considered the fuel of the energy transition, due to the possibility of generating future fuels, such as hydrogen (H_2). Currently, syngas is not very commercially attractive because, in addition to operating costs, it is mixed with carbon dioxide (CO_2) and other gases, thus, CO_2 must be separated from the syngas for commercialization. One economically viable alternative for purifying gases containing CO_2 is the Calcium Looping (CaL) process. CaL has several potential benefits for industrial applications, but the biggest challenge is to increase the capture capacity of the mineral sorbents (limestones) that quickly and progressively deactivate with use. Calcium-based sorbents doped with inert materials (MgO, Al_2O_3, ZrO_2, TiO_2, SiO_2, etc.) have been widely studied in recent years and are known as an efficient method for improving their capture capacity, however, their high preparation cost is their biggest disadvantage. In Brazil, energy generation from biomass burning produces large amounts of ash that generate environmental problems due to their final disposition. These ashes contain high levels of inert materials that can be used to dope limestones, but little is known about this. The article evaluated the performance of dolomitic limestones doped with rice husk ashes. The results indicate that ashes produced from rice husks have 96.33±0.08% of ash, 2.56±0.11% of volatile materials, and 1.74±0.05% of humidity. Loss on ignition indicates that the ashes have a mass loss of 5.19±0.28%, of which 4.30% refers to humidity and volatile material, and the rest is the volatilization of metals with low melting points. XRF analysis indicates that the ashes contain 94.76% of SiO2, 1.03% of CaO, 1.90% of K2O, 0.50% of Al2O3, 0.28% of Fe2O3, and 0.30% of*

Na2O and SO3. The dolomitic limestone used was ground and taken for analysis. The results indicate that it has 0.5±0.2% humidity, 38.21% CaO, 15.02% MgO, and 47.87% CO2 mainly. The cyclic tests of calcination/carbonation indicate that the dolomite doped with rice husk ashes performed better than natural dolomite. When compared to natural dolomite, the doped with CCA was better, probably due to the greater surface area provided by the rice husk ashes, and the high sintering temperature of the silica present in rice husk ashes.

Keywords: *calcium looping, sorbent, doping, biomass, sintering*

1. Introduction

Within the concerns regarding climate change mitigation, reducing greenhouse gas emissions in the atmosphere is one of the key objectives. One of the main gases, CO_2, has a significant percentage in emissions and can remain in the atmosphere for very long periods. According to the IPCC (2022), greenhouse gas emissions in 2019 were about 12% higher than in 2010, equivalent to 6.5 $GtCO_{2\text{-eq}}$. In 2010, CO_2 emissions had reached approximately 53 Gt. Furthermore, total CO_2 emissions in 2019 accounted for about 44.6 Gt, with emissions from energy consumption and industrial activities being the most significant. As a result, methods to solve this problem have been sought.

Thus, carbon capture and storage technologies (CCS) emerge as alternatives to minimize CO_2 emissions. Currently, there are three types of carbon capture systems: pre-combustion, oxy-combustion, and post-combustion (Wilberforce et al., 2019). Among the pre-combustion methods are physical absorption, adsorption, and membrane processes. Pre-combustion technologies are associated with systems that have a high-volume concentration of CO_2 in the stream (15% to 60%) (Theo et al., 2016) and essentially involve the reaction between fuel and O_2 to generate CO, H_2, and combustible gas. The oxy-combustion process involves fuel burning using pure oxygen, resulting in water vapor and CO_2 as combustion products (Falkenstein-Smith, 2017). Although pre-combustion and oxy-combustion are very promising, CCS technologies have mainly used post-combustion methods, such as amine scrubbing technology, which involves the chemical absorption of CO_2 by solvents composed of monoethanolamine (MEA) (Wilberforce et al., 2019).

Recently, other post-combustion methods used is called "Calcium Looping" (CaL) or Calcium Loop, based on the cycle of calcium carbonate formation. Basically, when CO_2 interacts with calcium oxide (CaO), it undergoes a reaction and forms calcium carbonate ($CaCO_3$). This reaction is known as carbonation. The reverse reaction of this process is called calcination (Blamey et al., 2010). During the CaL process, CO_2 passes through the carbonator, heated to around 580°C to 700°C (Erans et al., 2016), and interacts with the CaO-based sorbent used for absorption, resulting in $CaCO_3$. Then, the CO_2-loaded sorbent is transferred to the calciner, where it is subjected to temperatures between 900°C and 950°C (Erans et al., 2016), as the reaction is endothermic this requires external heat supply. In the calciner, a clean stream of CO_2 is generated, and the sorbent is regenerated to be taken back to the carbonator. CaL process is considered to be more advantageous compared to other post-combustion methods due to several factors: 1) the wide availability of limestone or dolomite (D) as a raw material for sorbents, which reduces operating costs; 2) established knowledge of carbonation and calcination reactions; 3) relatively low efficiency penalty, reaching less than 5% (Lockwood, 2017), whereas amine scrubbing technology has an efficiency penalty of 20% to 40% (Oh et al., 2018); 4) carbon capture cost of $16 - $44 per ton, compared to $34 - $80 per ton for amine scrubbing (Hongman et al., 2019); and 5) relatively low sorbent efficiency loss when compared with MEA. Additionally, considering that CaL process operates at high temperatures, there is the possibility of integrating the carbonation and calcination chambers with the combustion chambers, which represents a significant benefit (Abanades et al., 2005).

The major disadvantage with CaL process is the decay of sorbent conversion (capture capacity) after approximately 20 cycles of calcination/carbonation (Ortiz et al., 2019; Abanades, 2002). This decay is explained by the sintering process that occurs with CaO during calcination, where the sorbent is exposed to high temperatures. The Tammann temperature, which is the minimum sintering temperature of materials (Feng et al., 2017), for CaO is 1275°C, while for $CaCO_3$, it is 412°C (Daud et al., 2016). This sintering is typically associated with lattice diffusion (Perejón et al., 2016), where some atoms rearrange within the CaO structure, leading to pore obstruction and consequently reducing the sorbent particles' surface area (Perejón et al., 2016). To address this issue, the research has focused on incorporating inert materials with high Tammann temperatures into the sorbents to ensure sorbent stability even at high temperatures. Some of these materials include SiO_2, MgO, TiO_2, and Al_2O_3 (Daud et al., 2016). Another material with high stability and wide availability is SiO_2, whose Tammann temperature is higher than the operating temperatures of the calciner, and it can be found in biomass residues, such as rice husk ash.

For the other side, according to data from the FAO (Food and Agriculture Organization of the United Nations), in 2018, the production of rice amounted to 782.0 million tons. It is estimated that rice husk represents about 22% of the total mass of production. Rice husk is commonly used for energy production through direct combustion, pyrolysis, and gasification. During combustion, the primary method to energy production, a significant amount of ash is generated, primarily containing amorphous silica (on average, 90%). Due to composition of rice husk ash, this may environmental problems if disposed improperly. The use of rice husk ash as inert material for provide stability to the CO_2 sorbent can be made, but little is known about the subject. Thus, this article evaluates a viable alternative for utilizing of rice husk ash to increase the capture capacity of CO_2 sorbent by doping the sorbents, reducing sintering in the process.

2. METHODOLOGY

2.1 Materials

In this study, dolomite, named D, provided by Votorantim Cimentos company from the city of Itapeva, São Paulo state, was used. D was supplied in size 1 gravel, and it was reduced using a crusher and then a disc mill for approximately 5 minutes. The dolomite powder (D) was sieved and characterized. Rice husk ashes were produced in the laboratory using a biomass burner. They were named RHA. Once produced, they were ground and sieved for characterization. Both materials are shown in the Figure 1.

(a) (b) (c)

(d) (e) (f)

Figure 1. Samples of dolomite and rice husk ashes. (a) Dolomite (D), (b) Crushed D, (c) D after the grinding process, (d) Rice husk, (e) Rice husk ashes (RHA), (f) RHA after the grinding process.

2.2 Methodology of Calcium Looping:

The CaL process was performed using a thermogravimetric analyzer (TGA) from TA Instruments. The TGA was used to simulate cyclic calcination/carbonation tests. For the Calcination process, synthetic air was used. The sample was heated at a heating rate of 20°C/min from 30°C to 950°C. Once the temperature was reached, it was held for 5 minutes. Then, the sample was cooled to 650°C at a cooling rate of 20°C/min. Once the carbonation temperature was reached, the synthetic air gas was exchanged for 100% CO_2, and the temperature was maintained for 20 minutes. This procedure was repeated 6 times for each sample.

Figure 2. Simultaneous Thermal Analysis System Q600 SDT by TA Instruments.

2.3 Calculation of CO_2 capture capacity of the mixtures:

The effective conversion (X_{ef}) was calculated according to the methodology of Ávila I. et al. (2013). X_{ef} is the parameter used to evaluate the capture efficiency in each cycle of Calcium Looping (CaL). The effective conversion X_{ef} is defined as the ratio of the converted mass of CaO in the carbonation step of each N-cycle to the total mass of the sample before carbonation (Eq. (1)).

$$X_{ef} = \frac{Mass\ of\ CaO\ reacted\ with\ CO2}{Total\ mass\ of\ CaO\ in\ the\ sample} \quad (1)$$

$$X_{ef} = \frac{\frac{m_{CAR} - m_{CAL}}{W_{CO2}}}{m_A\left(\frac{Y_{Ca}}{W_{Ca}} + \frac{Y_{Mg}}{W_{mg}}\right)} \quad (2)$$

Where m_{CAL} and m_{CAR} are the masses of the samples after carbonation and after calcination in each N cycle, and W_{mg}, W_{Ca}, and W_{CO2} are the molar masses of Mg, Ca, and CO_2, respectively. Y_{Ca} and Y_{Mg} are the mass fractions of Ca and Mg in the natural dolomite (D) sample.

2.4 Preparation of dolomite - rice husk ash mixtures (D:RHA)

A total of 20g of each mixture was prepared. The D:RHA mixtures were prepared in the mass ratios shown in the table below:

Table 1. Rice husk ash in the samples (g).

Samples	2 %	5 %	10 %
Ashes mass (RHA)	0.4	1	2
Dolomite mass (D)	19.6	19	18
Mixtures total mass (D:RHA)	20	20	20

Once the samples were prepared, they were reprocessed in the disc mill for approximately 3 minutes to achieve granulometry homogenization and obtain a D:RHA mixture with uniform particle size (Figure 3).

(a) (b) (c)

Figure 3. Mixtures of dolomite and (a) 10 Wt.% (b) 5Wt.%, (c) 2Wt.% of rice husk ash

2.5 Characterization Methodology:

The samples of dolomite (D) and rice husk ash (RHA) were characterized by Proximate analysis and X-ray fluorescence (XRF) analysis to determine moisture (U), volatile matter (VM), ash content, fixed carbon (FC), and chemical composition of the samples.
Proximate Analysis: Immediate analysis was performed in triplicate on the RHA to determine their composition. For the calculation of moisture content, the samples were placed in crucibles and placed in an oven at a temperature of 105°C. After a period of 24 hours, the crucibles were removed and allowed to cool in a desiccator. Then, the masses of the crucibles with the samples were measured, allowing for the determination of the masses of the dried samples using Eq. (3). To determine the volatile matter, the dried samples were placed for three minutes in the open door of a muffle furnace heated to 850°C, and then placed inside the furnace for seven minutes with the door closed. After this step, the material was cooled in the desiccator, and the residual mass of the samples was measured and the % MV was calculated according to Eq. (4). Next, to determine the ash content (Ash %), the residual samples were placed in the muffle furnace at 500°C for approximately 4 hours. After this period, the crucibles with the samples were cooled in the desiccator, and their masses were measured, allowing for the calculation of the ash percentages. Using Equation 10, the ash content was determined. For the Fixed Carbon (FC %), it was determined based on the moisture content (U%), ash content (Ash %), and volatile matter (MV %), using Eq. (6).

$$U(\%) = \left(\frac{m_{umida} - m_{seca}}{m_{umida}}\right) \times 100 \tag{3}$$

$$MV(\%) = \left(\frac{m_{seca} - m_{resíduo}}{m_{seca}}\right) \times 100 \tag{4}$$

$$Ashes\ (\%) = \left(\frac{m_{seca} - m_{resíduo}}{m_{seca}}\right) \times 100 \tag{5}$$

$$CF(\%) = 100 - (MV\ (\%) + U\ (\%) + Ashes\ (\%)) \tag{6}$$

Chemical composition: The chemical composition of the samples was performed on powdered samples of dolomite (D) and rice husk ashes (RHA) using X-ray fluorescence (XRF). This analysis allowed for the determination of inorganic elements in the form of oxides.

3. RESULTS AND DISCUSSION

3.1 Results of material characterization

The characterization of rice husk ashes was realized and are presented in Table 2. Can be seen, the high ash percentage indicates a high concentration of inorganic material in the rice husk ashes.

Table 2. Immediate analysis of Rice Husk Ashes.

Parameters	%m/m
Moisture	1.7 ± 0.1
Volatiles	2.6 ± 0.1
Ashes	96.3 ± 0.1
FC	0.0 ± 0.1
Loss Of Ignition (LOI)	5.2 ± 0.3

From the XRF analysis, the percentage of each element within the rice husk ashes (RHA) was determined. It can be observed that the high percentage of SiO_2 (94.8%) agrees with the literature (around 90% of silica). Therefore, it can be said that rice husk ashes can be a promising source of SiO_2 for sorbent doping.

Table 3. Compositional analysis of Rice Husk Ashes (RHA) by element and their respective oxides. (%m/m)

Elements	Mg	Al	Si	P	K	Ca	Fe
	0.1 ± 0.01	0.2 ± 0.01	34.8 ± 0.6	0.4 ± 0.01	1.8 ± 0.20	0.8 ± 0.20	0.1 ± 0.27
Oxides	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	Fe_2O_3
	0.2 ± 0.1	0.5 ±0.1	94.8 ± 0.3	0.6 ± 0.1	1.9 ±0.1	1.0 ±0.1	0.3 ±0.1

The same XRF analysis was also performed on D. A significant content of MgO (15%) and 38.2% of CaO was observed. For the Calcium Looping process, these oxides are highly beneficial as they both form carbonates, and the addition of MgO in the sorbent aids in improving the porosity of small particles and reducing sintering (Daud et al., 2016).

Table 4. Compositional Analysis of Dolomite (D) by Element and Their Respective Oxides. (%m/m)

Elements	Mg	Al	Si	P	In	Ca	Fe
	9.3± 0.01	0.1± 0.02	1.5± 0.01	0.1± 0.01	0.4± 0.01	35.6± 0.02	0.2± 0.01
Oxides	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	Fe_2O_3
	15.0± 0.1	0.2± 0.1	2.9± 0.1	0.1± 0.1	0.4± 0.1	38.2± 0.2	0.2± 0.1

3.2 Chemical composition of the mixtures

With the aforementioned compositions (Table 3 and Table 4) and the mass proportions of the mixtures (Table 2), the chemical compositions of the mixtures, especially for the elements Ca, Mg, Si, and Al, were calculated and presented in Table 5. It is worth noting that many of the elements listed in Table 3 and Table 4 are not included in Table 5, due to these elements are present in very small quantities, and their amounts are not significant for the mixtures.

Table 5. Chemical composition of the D:RHA mixtures and natural dolomite (D). (%m/m)

	Ca	Mg	Si	Al
D (100%)	7.12	1.86	0.30	0.03
D:RHA (2%)	6.99	1.82	0.65	0.03

D:RHA (5%)	6.79	1.77	1.18	0.03
D:RHA (10%)	6.45	1.68	2.06	0.03

3.3 Results of CO_2 capture

The CaL tests were performed on both the D and their mixtures (D:RHA). The results of mass loss and the temperatures reached for each cycle in each sample are shown in Figure 4.

In the Figure 4, it can be observed that for all the mixtures, there is an initial mass loss of approximately 50% at the beginning of the process (first 50 minutes). This initial calcination is not considered in the efficiency calculations. The CO_2 mass loss of the samples ranges from 42% to 48%. The difference in CO_2 mass percentage among the mixtures is due to the heterogeneity of the D dolomite composition.

Furthermore, "peaks" can be observed in the mass variation (Black lines) with of course of cycles this peak represents carbonation reaction. After of each peak, there is the "valleys", which correspond to the mass of sorbent without CO_2. Since the second carbonation cycle onwards, the appearance of tips on the mass variation (Black lines) can be observed after carbonation reaction. These tips indicate a decrease in the diffusion-controlled stage, where carbonation occurs at a slower rate (Erans et al., 2016; Dean et al., 2011). Therefore, in the sharper peaks, the rate of carbonation per unit of time is higher. It was found that the carbonation occurred at faster rates in the D doped with 2% RHA compared to all other samples, while the D natural exhibited a carbonation process with more controlled diffusion.

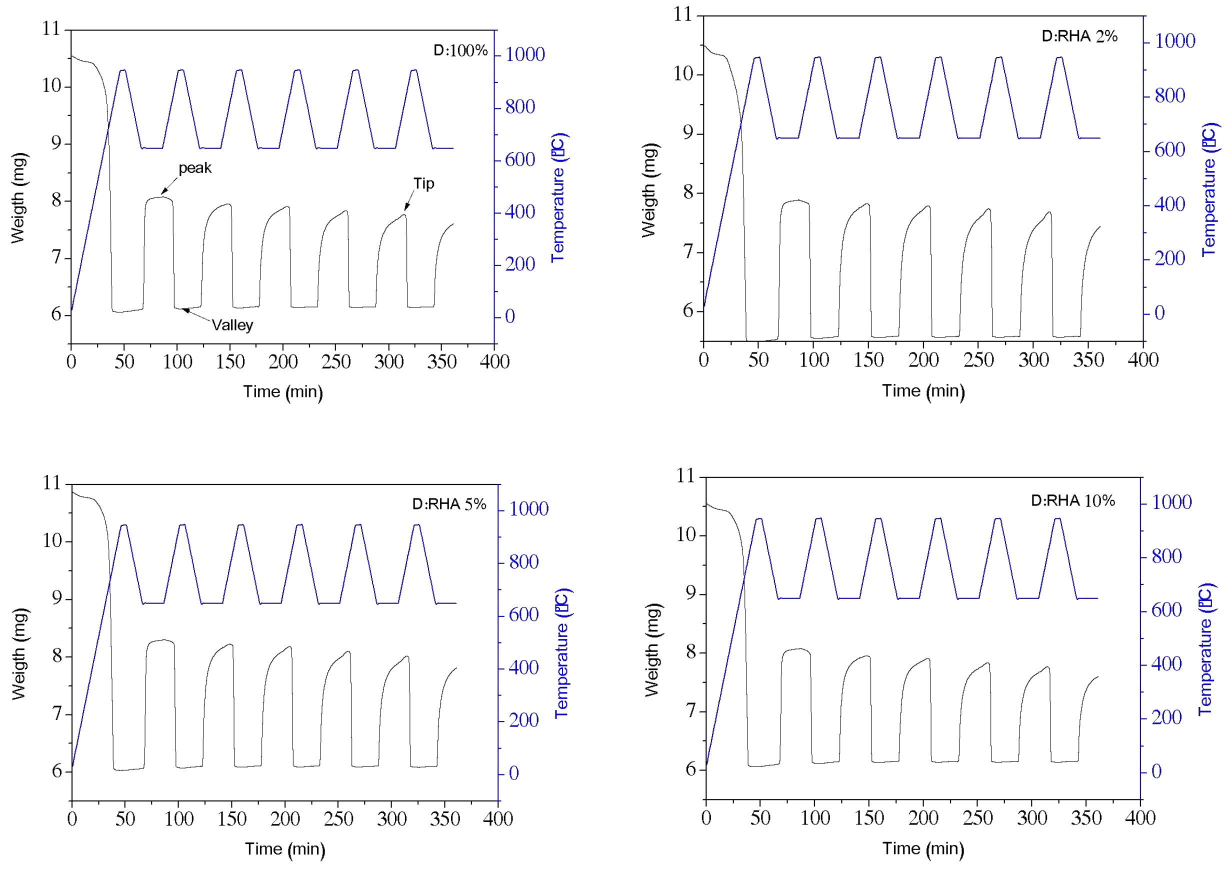

Figure 4. Calcium looping tests of Dolomite (D) and its mixtures (D:RHA) on the thermogravimetric balance (TGA).

Using the Figure 4, the capture efficiency X_{ef} was calculated for each cycle. The results of the calculation can be observed in Figure 5. It is interesting to note that the dry mixing process between dolomite and rice husk ashes (RHA) was able to modify the stability of the D dolomite. This is because increasing the percentage of RHA in the sorbent mixture reduces material sintering due to the high SiO_2 content in RHA, which has a higher sintering temperature than the operating temperatures within the CaL process. It can be observed that the efficiency lines of the mixtures doped with RHA had a lower degree of inclination compared to the efficiency line of natural dolomite (D), indicating greater sorbent stability.

On the other hand, despite assisting in reducing sorbent sintering, as the percentage of RHA in the mixture increases, there is a decrease in carbon capture efficiency. This is because with the increase of RHA percentage in mixtures results in a decrease in reactive CaO, thus the sorbent gains stability but loses CaO mass to react with CO_2.

Previous studies on doped sorbent with other materials such as Al_2O_3 and MgO, using mixtures with concentrations of 0%, 5%, and 10%, among other concentrations, indicated that the optimal carbon capture performance in the mixtures was at 5% (Sun et al., 2020; Benitez et al., 2018; Daud et al., 2016). However, in this study, it was observed that the sample with the highest stability and capture capacity during the 5 cycles performed was the mixture containing 2% RHA.

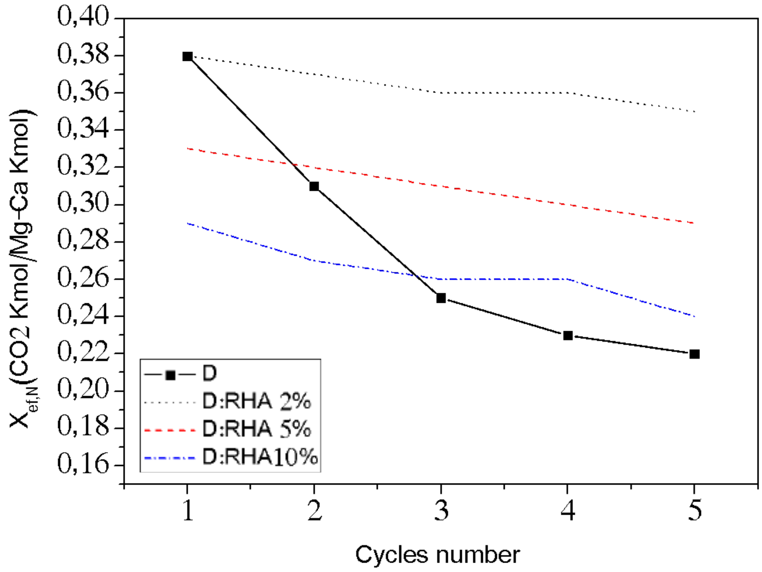

Figure 5. CO2 capture efficiency in each cycle for natural dolomite (D) and its mixtures (D:RHA).

4. CONCLUSION

Therefore, based on the observed results, it can be inferred that currently, rice husk ash can be a promising source for the supply of SiO2 for sorbent doping in Calcium Looping, as the ash is a residual byproduct and therefore low-cost. Additionally, SiO2, which is responsible for reducing sintering, is the main compound in rice husk ash, accounting for around 90%, indicating high performance in improving sorbent stability.

By using mixtures with different proportions of rice husk ash, such as 2%, 5%, and 10%, an improvement in CO2 capture stability during the carbonation and calcination cycles was observed. Furthermore, it was noticed that increasing the ash content in the mixture resulted in a loss of CO2 capture capacity. This is because an increase in the percentage of SiO2 in the mixture leads to a reduction in the percentage of CaO, thereby decreasing the reactivity of the mixture.

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