

INVESTIGATION ON PRODUCING SILICA FROM RICE HUSK BIOMASS

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ABSTRACT

Intending to contribute with the valorization of rural squanders that present transfer issues and the utilization of sustainable power sources, was looked into the rice husk burning in climatic foaming fluidized bed reactor. The technique was to assess the air overabundance impact in the pipe gases and the powder attributes. It was resolved the gases organization (CO, CO₂ and NO_x) and portrayed the ash produced (XRD, XRF). 40% excess air advances temperatures inside the reactor around 700°C with higher change efficiencies but declining the nebulous silica capability of the fiery debris. Opposite conduct was confirmed at 128% overabundance air. The pipe gases demonstrated relative inconstancy for each working condition. In the CO case, a normal grouping of 200 ppm was found (on a dry premise of 11% O₂) for air overabundance somewhere in the range of 40.0% and 82.5%.

Keywords: rice husk, rice husk ash, combustion, fluidized bed, silica

1. INTRODUCTION

In Bangladesh, according to Agrocadenas MRD et al. (2008), approximately 520 thousand annual tons of rice husk (RH) are produced and its disposal is the main environmental problem facing the rice industry. The main components of the AC are cellulose and hemicellulose (50%) in addition to lignin (26%) and organic components such as oils and proteins (4%). Approximately, the remaining 20% includes different substances of inorganic character such as SiO₂, Al₂O₃, K₂O, Na₂O, MgO, CaO, Fe₂O₃, MnO, P₂O₅. Rice husk ash (RHA) is the solid residue of any thermochemical transformation (pyrolysis, gasification or combustion) developed from the RH. Their physical-chemical characteristics depend mainly on the conditions

implemented in the process. The RHA corresponds to 14-25% of the AC depending on the region's rice, climate and soil variety by Krishnarao et al., (2001). The main component of the RHA generated at moderate combustion temperatures is amorphous silica in compositions comprised between 80 and 97% by Chandrasekhar et al., (2005) & Yalçin et al. (2001). On the other hand, bubbling and atmospheric fluidized-bed reactors (RLFBA) are considered as a viable and effective method for the development of different operations and treatments involving mass and energy transfer. This technology allows the thermochemical transformation of heterogeneous materials of irregular shape and low density.

As the RH, given its inherent advantages in terms of the flexibility of the material to be treated, low temperatures and isothermal conditions throughout the reactor. In this sense, several studies on gasification and combustion of rice husk in this type of reactor can be found in the literature as reported by Barriga et al. (2002) Armesto et al. (2002) Fang et al. (2004) Cortês et al., (2004) Ramírez et al., (2002). Okasha et al. (2007), Andrade et al. (2007), Rozainee et al. (2008), Singh et al. (2008), Vélez et al. (2009). On the other hand, the studies to obtain silica from this biomass have been concentrated in laboratory equipment in a fixed bed, where high reaction times and a considerable consumption of electricity are necessary, as reported by Yalçin et al. (2001), Liuo et al. (2004), and Della et al. (2005).

Based on the above, the combustion of RH in RLFBA at moderate temperatures can be consolidated as an attractive alternative for the generation of hot gases with a low content of pollutants, with potential in the different industrial drying processes, such as the rice farmers. The present work shows the results of the combustion of RH with high excess air in a pilot scale RLFBA. The results obtained allow us to conclude on the effectiveness of the

process studied, in terms of the production of hot gases and silica rich ash with high potential for other industrial processes.

2. METHODOLOGY/ANALYSIS/EXPERIMENTAL SET-UP

The RH used in the combustion experiments was acquired at Bangladesh Rice Research Institute (BRRI) lab, Bangladesh with the collaboration of CONACYT Lab in Latin America. The RH originated in the city of Ibagué-Tolima and was not processed or modified for the combustion experiments. Table 1 shows the elementary, immediate and calorific value analysis on dry basis of the RH used in the research.

Table 1 Elementary and immediate analysis on dry basis of the RH

Elementary Analysis	%	Immediate analysis	%
Carbon	36.6	Volatiles	57.7
Hydrogen	5.83	Humidity	9.3
		Residual	
Nitrogen	3.31	Ashes	17.6
Oxygen	36.65	Fixed carbon	15.4
Sulfur	0.01	Higher calorific power (MJ/kg)	14.61

2.1 Fluidized bed reactor

The equipment used for the combustion of the RH was an BRRI Lab, located in the Bangladesh, and designed by researchers from the IUT, University by Ramirez et al. (2007). The reactor has a reaction chamber of 0.3m internal diameter, with expansion to 0.4m in the freeboard area and 3m height. Likewise, it has a silo with capacity for the storage of approximately 1,400kg of RH, as well as a system of threads in charge of dosing and feeding the material to the reactor. A general scheme of the equipment is shown in figure 1.

2.2 Burning rice husk in bubbling fluidized bed and atmospheric

Initially 40kg of sand was introduced into the fluidized bed reactor in order to obtain have a static bed height of 0.4m. Subsequently, the industrial burner was switched on, until the inert bed reached an average temperature of around 400 ° C. Once the ignition temperature of the scale is reached, the feed system is activated by setting stoichiometric combustion conditions ($\approx 3.6\text{Nm}^3 / \text{kg}$) until the temperature inside the reactor reaches 750 ° C. Finally, the conditions of excess air (EA) desired for the experimentation are adjusted by means of the AC discharge, which was controlled in turn by a frequency inverter (VFD) coupled to the motor that allows the movement of the thread do- fluidization rate (U_f) was controlled thanks to the readings of the flow indicator

(Magnetrol TA2 differential heat meter) and the manipulation of the valve located after the blower. The operating conditions were 40%; 82.5% and 125% excess air, setting in all cases a U_f of 0.14 Nm / s.

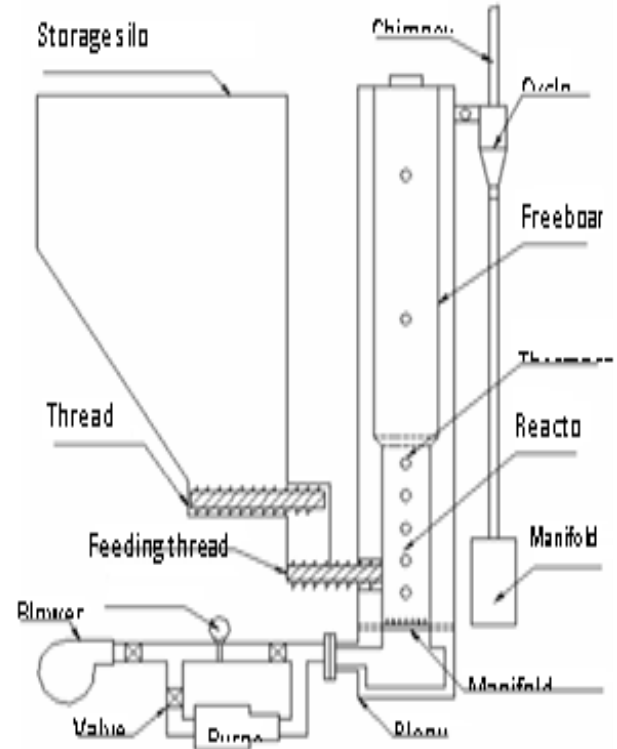


Figure 1: Schematic of the atmospheric and bubbling fluidized bed reactor

2.3 Characterization analysis

Once the permanent regime was reached, based on a stable temperature inside the reactor, measurements of CO, CO₂ and NO_x were made by means of a portable analyzer BACHARACH brand NSX300 reference of electrochemical sensors. The data corresponding to the temperature were obtained by type K thermocouples located in the body of the reactor. The data was captured using an ADAM reference data acquisition system reference 4118 and stored on a PC through the SMILES VIEWER® software. On the other hand, the RHA obtained was characterized for each experimental condition, by means of X-ray diffraction (XRD) and X-ray fluorescence (FRX) techniques. For the DRX analysis, a PANalytical brand diffractometer was used, model XPERTPRO-MPD, with radiation over α , power of 45kV and current of 40mA in order to know the crystalline character of the ash. The analyzes were carried out on dust samples in the 2 θ measurement range between 15 and 70° and developed in the Materials Characterization Laboratory of the National University of

Colombia (UNALMED), Medellin. The FRX analyzes were performed on a PHILIPS equipment, model PW 2400 with 3kW tube. The tests were carried out in the instrumental analysis laboratory of the private company of Corona Group, SUMICOL S.A.

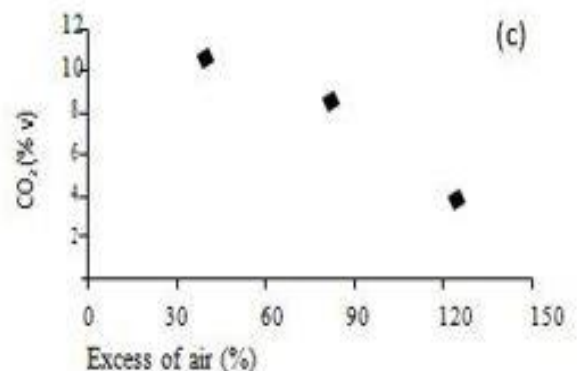
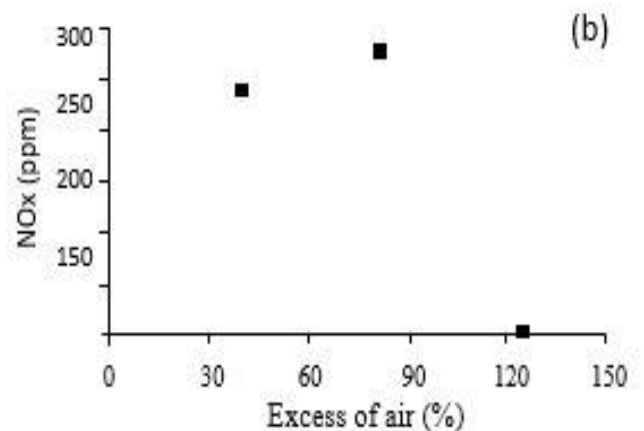
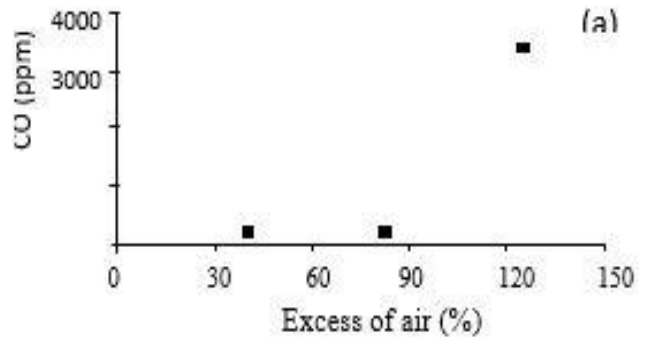
3. RESULTS AND DISCUSSION

According to experiments previously carried out, the fluidization velocity implemented in the process (0.14 Nm / s) guaranteed the development of fluidization, for the RH and sand mixture used. Similarly, and according to by Fang et al. (2004) and Qiaoqun et al. (2005); high values of U_f favor the decrease of the phenomena of segregation in the bed, obtaining a better distribution of the materials throughout the reactor, as well as better mixing conditions. Low U_f correspond to longer residence times of the particles, allowing better conditions for the transformation of the organic material present in the husk by Cortês et al. (2004). On the other hand, lower EAs lead to higher values of temperature in the bed, promoting the transformation of amorphous silica into crystalline by Rozainee et al. (2008) and Della et al. (2006). In figure 2, the emissions in dry base of CO are presented, CO₂ and NO_x for the experimental conditions implemented, based on an O₂ concentration of 11% (resolution 0058 of 2002 of the MA-VDT, article 25); as well as the temperature middle of the bed. In the same way in figure 3, the conversion efficiency of the AC is shown in function of the air excesses implemented. The conversion efficiency of the RH (η) was calculated based on the formula presented by Armesto et al. (2002), as shown in equation (1). The terms involved in this equation correspond to an energy balance in order to determine the conversion of the carbon present in the AC. ERH is the energy corresponding to the shell, ERHA the loss energy for the residual carbon present in the RHA, and ECO the loss energy corresponding to the CO in the gauge stream that leaves the reactor. The calculation of ERHA, considered the percentage of ash reported in the immediate analysis based on the mass flow of RH fed to the process, as well as the values of ignition losses that are reported in table 2.

$$\eta = \left(\frac{Erh - Erha - Eco}{Erh} \right) 100\% \quad (1)$$

The results presented in figure 2 show that air excesses in the range between 40% and 82.5% have tolerable concentrations of CO around 200 ppm (on adry 11% of O₂). However, increases in air to the upper limit (125%) showed a considerable increase in concentration of this compound. This behavior is expected and is attributed to the decrease in the temperature of the bed (Figure 2d), which discourages complete transformation of the carbon

present in the RH into CO₂. This trend was also observed by Okasha by Okasha et al. (2007), in rice straw pellet combustion experiments in RLFBA. Similarly, the decrease in NO_x concentration (from 317ppm to 6.27ppm in a dry base of 11% O₂) is in line with the performance of the graph is expected. Increases in the amount of oxygen fed to the process, disadvantage the formation of NO_x by thermal mechanism, given the low temperatures reached (Figure 2d), compared to the values of temperature of formation of the compound, which begins to be considerable around 1100 °C.



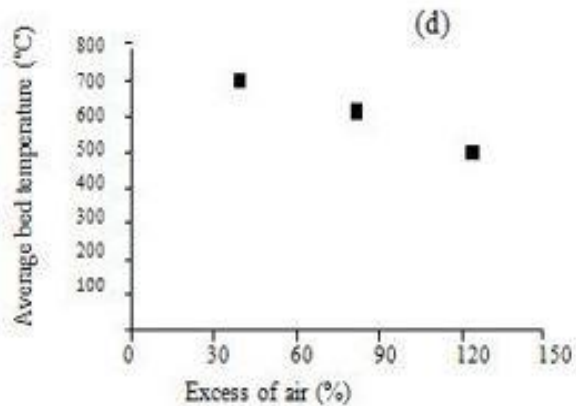


Figure 2: a) Emissions of CO (ppm) at 11% of O₂, b) Emissions of NO_x (ppm) at 11% of O₂, c) Emissions of CO₂ (% v) at 11% of O₂, d) Average temperature of the bed (°C) depending on the excess air of oxygen in the process, cause lower values of reaction temperature causing a lesser use of the energy contained in the biomass.

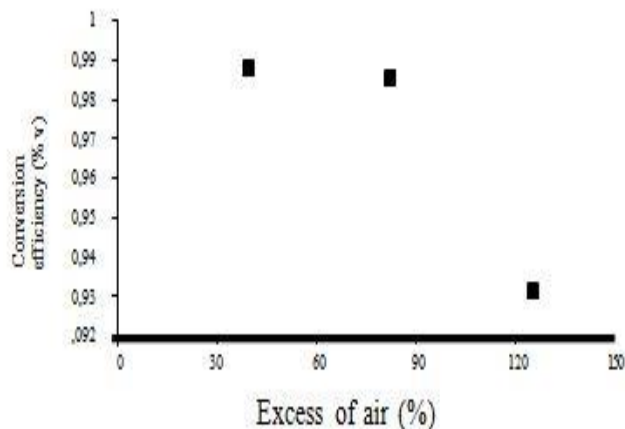
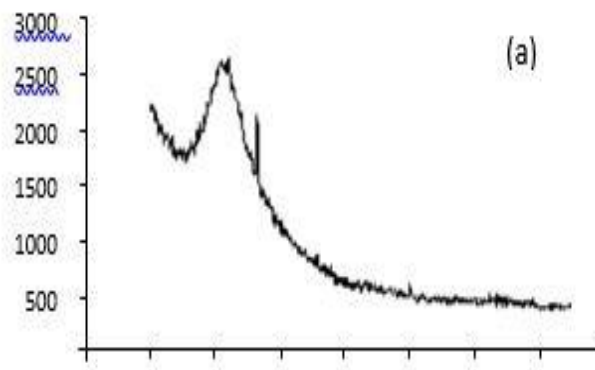


Figure 3: Efficiency of thermo chemical transformation of the CA in RLFBA with U_f of 0.14 Nm / s, depending on the excess of air.

In the case of transformation efficiency, values around 98% can be reached at air excesses of up to 82.5%. Larger amount of oxygen in the process, cause lower values of reaction temperature causing a lesser use of the energy contained in the biomass. The trend shown in Figure 3 is consistent with the behaviors found for bed temperature and CO emissions. The thermo chemical transformation efficiency should not be confused with the overall efficiency of the process, which usually considers the energy of loss per walls.

3.1 X-ray diffraction

Through the XRD analysis it was found that the solids obtained in the combustion process, for the different air excesses implemented, they have an amorphous predominance, that is, with a structure formed by atoms with a short distance orientation, highly reactive and easy to process. In Figures 4a, 4b and 4c, the respective diffractograms are presented to the conditions of EA worked. In the case of the diffractogram of the RHA obtained from the combustion condition of 40% of excess air (Figure 4a), three important peaks are evidenced, standing out the one located at 22 ° which indicates the presence of the amorphous phase. of silica in the RHA. The two remaining peaks, of medium intensity, located at positions 26.63 ° and 50.1 ° are associated with small amounts of crystalline silica, given the temperature reached in the bed (700 ° C, figure 2d). According to Della et al. (2005), the temperature and time of oxidation of the RH are important factors that govern the properties of the silica contained in the RHA, certainly the SiO₂ content, its amorphous or crystalline nature and consequently its reactivity. For excess air around 82.5%, when the average temperature inside the reactor is around 606 ° C (Figure 2d), a decrease in the intensity of the peaks at positions 26,63 is observed. ° and 50.1 °, as it is observed in figure 4b. Different researchers such as Armesto et al. (2002), Rozainee et al. (2008) and Della (2005) evidenced transformation of amorphous silica into crystalline, specifically in the form of cristobalite, from high temperatures and prolonged periods of time. According to the authors, the crystallization of the silica of the RH is characterized first by the formation of crystals of cristobalite and then of tridimite, influenced mainly by temperature. For excess air around 125%, when the average temperature inside the reactor is around 500 ° C (Figure 2d). Only the presence of the amorphous phase with a broad base peak corresponding to 22 ° is appreciated, without any considerable peak showing the presence of crystals in the RHA (Figure 4c).



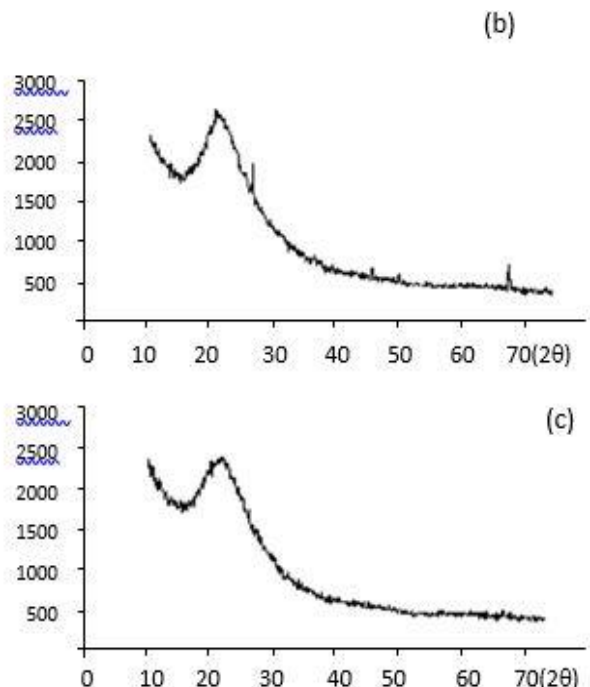


Figure 4: Diffraction pattern for the obtained RHA. (to) for the 40% EA condition, (b) for the 82.5% EA condition, (c) for the 125% EA condition.

3.2 X-ray fluorescence

According to the FRX analysis, presented in Table 2, the chemical composition of the RHA obtained in the combustion experiments for the working conditions implemented, showed as main component the SiO_2 with values around 92% by mass (for air excesses of 40 and 82.5%), followed by K_2O (<2.3%), P_2O_5 (<0.8%) and CaO (<0.5 %). In general, and according to the results of. In the case of science, the results obtained showed that increases in the EA disfavor the transformation of the carbon present in the husk, giving rise to a higher concentration of burned in the ashes. In relation to inorganics (SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , Na_2O , K_2O , Cl , SO_3 , MnO , P_2O_5), mainly for silica (SiO_2), lower excesses of air (and consequently higher temperatures of reaction) favor greater amounts of the compound in the ash, precisely because of the decrease in the amount of carbon in it. Experiments to obtain silica from AC in an electric oven at 700°C and 4 hours of calcination, with high and low heating rates developed by Krishnarao et al. (2001); they reported amounts of carbon, silica (SiO_2) and potassium (K_2O), around 4.2; 90.0 and 2.0% respectively. The researchers highlight the effect of potassium in the fixation of carbon in the ash, due to the dissociation and melting of it at relatively low temperatures, 620 and 336.8K respectively. In this sense, and considering the energy self-sufficiency of the combustion of RH in RLFBA, the continuity of the process in relation to the production of ash with silica content predominantly amorphous with quantities of

carbon (small ignition losses) (around 3.0% for excess air around 82.5%) the effectiveness of the thermo chemical conversion of RH in fluidized bed reactors is demonstrated.

Table 2: Chemical composition of the RHA

Component	40% of SHE	82.5% of EA	125% by EA
SiO_2	93.3	92.1	83.6
Al_2O_3	0.15	0.26	0.16
Fe_2O_3	0.14	0.2	0.088
CaO	0.38	0.42	0.45
MgO	0.31	0.33	0.32
Na_2O	0.01	0.01	0.01
K_2O	2.2	2.25	2.08
Cl	0.23	0.27	0.17
SO_3	0.24	0.26	0.06
MnO	0.18	0.17	0.21
PO	0.64	0.7	0.76
PPI (1)	2.6	3	12.6

4. CONCLUSION

Greater air excesses allow lower reaction temperatures, lower efficiencies of thermo chemical transformation of the AC, and lower amounts of silica in the RHA. However, the amorphous character of the solid obtained (RHA) is favored according to the lower amounts of crystalline silica thanks to the effect of the low bed temperatures. The results found from the experimentation of developed, demonstrate the technical feasibility of valorize an agro-industrial waste like RH in obtaining energy in the form of heat, useful in different industrial drying processes. Similarly, the process implemented showed potential in the sustainable obtaining of ash with high contents of amorphous silica (> 93%) and low carbon content (losses due to ignition) of less than 3.5%. In this sense, the potential of this type of reactors in the obtaining of rice husk ash rich in amorphous silica with respect to continuity and self-sufficiency was demonstrated. The energy science of the process, compared to the methods usually investigated in bedfixed, which consider external sources of energy and high reaction times. The RHA generated in the combustion process in RLFBA, can serve as the initial base in the obtaining of raw material with potential in the ceramic industry (to produce pigments or abrasives), in

the construction area (as a pozzolanic material) or to produce electronic parts.

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