

Valorization of rice husk chars as adsorbent: characterization and utilization in a novel reactor operating mode

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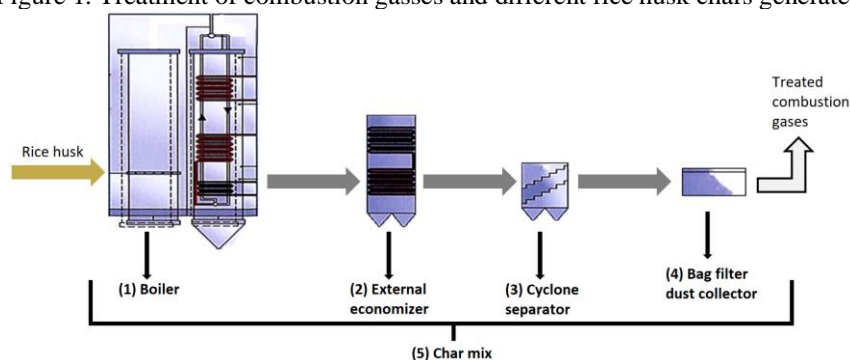
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The incineration of rice husk produces a residue, rice husk char, that is an environmental problem. The char is generated at the steam boiler (1). However, it is also removed during the treatment of the combustion gasses as they blow away char particles (Figure 1). Rice husk chars are gathered at the external economizer (2), the cyclone separator (3) and the bag filter dust collectors (4). (5) corresponds to the mix of all previous chars. The properties and composition of the chars could be different but literature regarding those differences has not yet been found.

Figure 1. Treatment of combustion gasses and different rice husk chars generated.



Rice husk chars have been used as adsorbent to remove undesirable compounds from wastewaters (Gupta and Suhas, 2009). The wastewaters of the rice mill industries have contaminant compounds that can be removed by adsorption. As reported in literature, phenol is one of those compounds (Kumar *et al*, 2016). Rice husk char could be a substitute for commercial adsorbents.

The reduction of energy and resources consumption is a key aspect in process sustainability policies. An ideal process uses the least amount of energy in transport, material movement and mixing, as well as the minimum amount of resources, especially if these are imported or expensive. Adsorption processes are widely used to remove compounds from wastewaters. There are different adsorption reactors, being the most common fixed beds and stirred tanks (Seader *et al*, 2010). Stirred tanks are operated in batch mode, in which solid and liquid are added into the tank and completely removed after a set amount of time; continuous mode, where adsorbent and liquid are continuously added and removed from the tank; and semicontinuous mode, where adsorbent is retained in the tank, but liquid enters and exits the tank continuously. Most of these modes throw away adsorbent that has unused adsorption capacity. Regarding the sustainability policies, the reuse of the adsorbent could be of great interest.

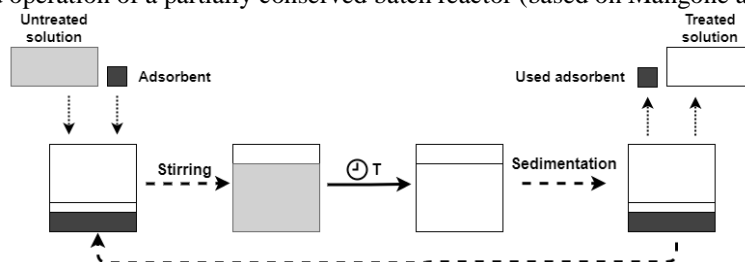
Considering all previous aspects, this work proposes the use of rice husk chars as adsorbent to remove undesired compounds from wastewaters. Particularly, we focus here on wastewaters from rice mill industries. The work is organized in two sections. The first one is related to the characterization and evaluation of adsorption properties of the different chars generated at the industrial plant. The second one compares operation parameters and performance of a proposed adsorption reactor operation with a traditional adsorption reactor operation.

Characterization of rice husk chars: Rice husk chars from the points indicated in Figure 1 are sampled and characterized. Density, particle size distribution, thermogravimetric analysis, elemental analysis, X-ray diffraction (XRD), and N₂ adsorption-desorption isotherm are some of the characterization techniques used. To evaluate the adsorption capacity of the chars, adsorption experiments are carried out using methylene blue and phenol as adsorptive. Methylene blue is used to determine the chars with better adsorption potential. The selected chars are then used to study the adsorption of phenol from a water solution. Adsorption kinetic is evaluated at three initial concentrations of phenol using the selected chars as adsorbents. Pseudo-first order (PFO) and pseudo-second order (PSO) kinetic models are compared to the experimental data to determine the best fit, and kinetic parameters are calculated. Phenol adsorption and adsorption kinetic are also studied with activated carbon for comparison.

Results show that density value increases in the order (2), (3), and (4), while mean particle size decreases in the same order. The latter is expected as the smallest particles are removed in the bag filter dust collector. Thermogravimetric analysis shows that each char has different organic matter content. Although most chars have less than 10% (m/m) of organic matter, the bag filter dust collector char presents a value near 25%. Elemental analysis shows a similar tendency on carbon content, being 17% (4), and between 2.8% and 7.6% (rest of chars). When SiO₂ crystallinity is analyzed by XRD, (1) presents crystalline SiO₂ as cristobalite, while the rest of the samples present a higher proportion of amorphous SiO₂. Finally, BET areas are calculated from N₂ adsorption isotherms and the areas for each char, in m².g⁻¹, are the following: 11 (1), 56 (2), 54 (3), 45 (4), and 35 (5). The characterization of the rice husk chars shows the chars have differences between each other. The analyzed properties suggest the char obtained from the dust filter dust collector should have the greatest potential as adsorbent from all chars. In the adsorption experiments, it is observed that (4) presents the higher adsorption capacity for methylene blue (56.5 mg/g) and phenol (13.4 mg/g). This result confirms the previous findings regarding characterization. While studying the adsorption kinetics, all analyzed chars an activated carbon reach equilibrium at similar times, being (4) the char that presents the shortest time. It is determined that PSO presents the best fit for all chars and activated carbon, at the different initial concentration of phenol studied. Experimental results and kinetic parameters are presented.

Operation of an adsorption reactor: A novel operation strategy for a stirred tank reactor, called partially conserved batch reactor (PCB), is proposed. PCB operates in batch mode with partial removal of adsorbent and liquid from the reactor at the end of an operation cycle (Figure 2). During the next cycle, a small portion of fresh adsorbent is added to the tank, as well as untreated solution. This allows to take advantage of the unused adsorption potential of the adsorbent that is already inside the reactor. The total adsorbent mass and liquid volume remains unchanged from one cycle to the next. After several cycles, it is observed that the system reaches a periodic behavior in which the final adsorptive concentration of one cycle is equal to the final adsorptive concentration of the next cycle.

Figure 2. Proposed operation of a partially conserved batch reactor (based on Mangone and Gutiérrez, 2020)



Relevant operation parameters in this model are adsorbent mass (m), adsorbent mass removed at the end of a cycle (m_e), liquid volume (V), liquid volume removed at the end of a cycle (V_e), initial adsorptive concentration (C_0) and adsorption time (T). Kinetics parameters are also needed to describe the adsorption process. Different cases are presented in this work to compare the removal efficiency, required time and adsorbent mass requirement between PCB and other operation modes when an adsorptive concentration is required at the end of the adsorption process. As an example, for a set of operating parameters, PCB and batch operation are compared. It is observed that PCB reduces the amount of adsorbent used nearly ten times compared to a batch operation, when the same volume of wastewater is treated.

In conclusion, rice husk chars show adsorption properties which vary depending on the equipment where the char is generated. The bag filter dust collector char presented the best adsorption capacity for methylene blue and phenol. Finally, the proposed operation for a stirred tank reactor allows to better select the operating conditions that leads with the trade-off of maximizing adsorbent potential while minimizing adsorption required time and reactor volume.

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