Synthesis, characterization and application of biochar from dyeing sludge in the removal of dyes

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In municipal wastewater treatment, the disposal of generated sludge is a major problem and the operating cost of sludge treatment can reach approximately 50% of the operating cost of the station. In wastewater treatment of textile industry, sludge is generated by biological or physicochemical treatments, causing high disposal costs for industrial laundries. Normally, the sludge is pressed and taken to landfill for disposal. The fashion industry is of major relevance to the world economy, worth $ 1.3 trillion and employing 300 million people along its chain. It can be considered one of the largest in the world by the size of production and the number of jobs created. Brazil has the fifth largest textile industry in the world, a sector of great importance in the Brazilian economy. In 2017 is generates 51.8 billion dollars income and more than 1.5 million direct jobs across the country. This industry is characterized by requiring large volumes of water and generating a large amount of effluents consisting of suspended solids, non-biodegradable organic matter, dyes and a range of chemicals. In general, for the treatment of these effluents in the textile industries is applied the biological treatment via activated sludge systems, followed by physical-chemical coagulation, flocculation, decantation and filtration processes. Activated carbon is an efficient means for treating industrial effluents, as it has high adsorption capacity, but is still considered an expensive product for large scale application. This porous carbonaceous material, which has a high internal surface, can be obtained from numerous organic materials after calcination. The removal of dyes from industrial effluents by natural adsorbents has been widely investigated, given the potential of natural adsorbents for this purpose (Jarusiripot, 2014). This work aims to synthesize and characterize a biochar produced from the pressed sludge collected in an industrial laundry and apply in the treatment of its own industrial effluent. The textile sludge used was collected from physicochemical treatment plant of a Brazilian industrial laundry.

For the production of biochar, the pressed sludge was disposed in a 18 L metal cans opened and heated for 6 hours to dry the material in a 2x1.5x1m wood-fired furnace. Then the cans were sealed and charred for 2 hours. After this period, heat dissipation was awaited until room temperature and then opened. The use of the furnace for synthesis of biochar was intended to simulate the use of boilers already existing in the industrial laundry.

The biochar was characterized for ash, volatile and fixed carbon analysis, particle size analysis, pH, nitrogen adsorption/desorption analysis, Boehm titration, X-ray dispersive energy scanning electron microscopy (SEM EDX), elemental analysis and Point of Zero Charge (PZC). Dry textile sludge was also characterized for ash content, volatile and fixed carbon analysis, elemental analysis and nitrogen adsorption/desorption analysis.

Batch tests were performed to observe the behavior of biochar as adsorbent in a real industrial effluent solution from the textile dyeing process. Biochar dosage and pH were varied to verify the changes in the adsorption kinetics, turbidity, color and chemical oxygen demand (COD). All experiments were performed at room temperature.

The average moisture content in the sludge samples was 79.21%, a very high value despite being pressed, there is a lot of water trapped in the material. Biochar presented 28.77% of fixed carbon, 17.6% of volatiles and 38.1% of ash. SEM-EDX data with elemental mapping indicated the elemental composition and distribution of oxygen, manganese, silicon, aluminum and magnesium on the biochar surface. A high percentage of aluminum (14.6%) was detected, and this should be due to the aluminum polychloride coagulant added to the wastewater treatment. Manganese and potassium correspond to potassium permanganate used in the textile processing process. The biochar presented a total acidity of 3.93 mmol g^{-1}, being prevalent the carboxylic groups, followed by lactonic and phenolic. From the particle size analysis of biochar, it was observed that more than 70% of the material has a particle size greater than 0.212 mm and is considered a coarse material. The pH of the biochar measured after 24 h stirring in water was 6.3 and the pH of the distilled water measured at the beginning of the experiment was 5.8. This pH increase may be related to basic functional groups on the charcoal surface. The PZC of the biochar was 6.7. The nitrogen adsorption/desorption isotherms of textile sludge and biochar have a similar profiles, with type IV of the IUPAC classification, characteristic of a micro and mesoporous material. The BET surface areas of sludge and biochar were 70 and 176 m^2g^{-1}, respectively. According to literature data (Kaçan, 2106), the surface area obtained for biochar without any activation can be considered high.

Leaching tests performed by contacting the sludge and biochar with an acetic acid solution showed that there was a greater turbidity of the solutions at the end for the sludge tested. Sulphate, free chlorine, Mn, Cu, and Fe species showed higher stability in biochar, with low leaching rates.

The results of turbidity reduction, color (at 452 nm) and COD for industrial effluent after testing with different doses of biochar are shown in Figure 1 (left), while the influence of pH in a treatment with 0.65 g of biochar in 40 ml of effluent is presented Figure 1 (right). According to the data, the adsorption rate reached the optimal point for concentrations between 0.5 g and 0.8 g of the adsorbent to 40 ml of sample, with a color and turbidity reduction of 95% and 50% COD.

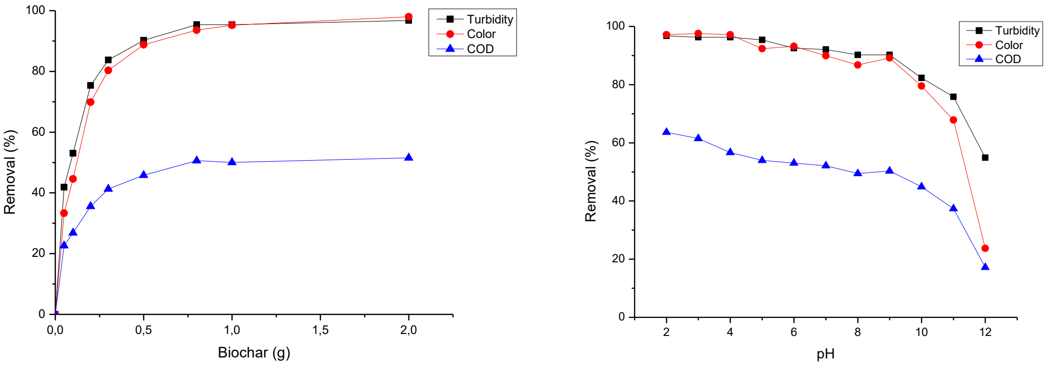

Figure 1. Turbidity, color (452nm) and COD removal as a function of biochar mass (left) and pH (right).

The best adsorption results occurred in acidic conditions, at pH below 6 where the COD reduction ranged from 53 to 63% and the color removal and turbidity remained between 92 and 97%. For pH values above 6, the removal rate dropped quickly, resulting in only 17% COD, 55% turbidity and 23% color removal at the pH 12. These results match the biochar's PZC of 6.7, since at pH values below that, the biochar is positively charged facilitating the adsorption of negative species such as reactive dyes that are anionic.

A kinetic study showed that after 30 min of the test COD removal stability state reached 50% and the turbidity and color removal reached the equilibrium after 2 h with 92% removal. COD stability occurs in a quarter of the time of color and turbidity equilibrium, therefore for the biochar application in a treatment system a time greater than 30 min would be the optimal.

The sludge carbonization process is also interesting because, in addition to decreasing the volume of the material, it increases the stability of metals, mitigating the risks of this polluting source in the environment, as well as valorizing the residue that can be applied as adsorbent, for example. This biochar could be produced inside the industrial laundry itself, using as well as a heat source for burning the boilers.

The conversion of these textiles residues into carbon for use as an adsorbent in wastewater treatment is a trend and a promising alternative from the economic, environmental and social point of view, and the number of studies in the literature on the synthesis of biochar from textile sludge to wastewater treatment is limited.

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