

Production and Characterization of Rice Husk Biosorbent from Far North Cameroon

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Abstract

The objective of this study is the production and the characterization of rice husk biosorbent. In fact, the biosorbent has been obtained by phosphoric acid treatment; its physicochemicals characteristics such as point of zero charge, specific surface, iodine number and chemicals functions have been determined. The analysis indicated that, the point of zero charge is 8.7; for the pH value less than 8.7, the biosorbent surface is positively charged and for pH value higher than 8.7, the biosorbent surface is negatively charged. The biosorbent iodine number is 1560.87 ± 1 mg/g, it means that, the biosorbent is constituted in majority of microspores. Furthermore, the specific surface of biosorbent is 104.45 ± 1 m²/g, it is five times as big than untreated rice husk obtained by Dada and al., (2012). Acid treatment improve the porosity of biosorbent. Infrarouge spectrum present ether and aromatic functions.

Keywords: rice husk, biosorbent, by phosphoric acid treatment, characterisation, Far north-Cameroon

1. Introduction

Activated carbon is the most adsorbent used in adsorption process due to its extend surface area, its microporous structure and its high adsorption capacity. However, its higher cost specializes the research to low costs adsorbents. Different biomass has been test as low-cost adsorbent (Özacar et Sengil, 2005; Saeed et al, 2005; Ho, 2005; Kumar et Kumaran, 2005; Franca et al., 2008; Dávila-Jimenez et al., 2009; Dada et al., 2012; Tchuifon, 2016, Eko, 2016; Mouthe, 2017). In the Far North Region of Cameroon, important quantities of rice husk (60000 tonnes) are product each year. These waste solids provided from rice growing practice in that Region and represent 20 % of the whole rice (Daifullah et al., 2003). In spite of their abundance, these rice husks are abandoned in the nature and polluted the environment. The advantages of the use of these waste solids are:

- ❖ The valorization of agro resources which are available, and low cost;
- ❖ The positive repercussions of activated carbon cost;
- ❖ The creation of geriatric activities jobs;
- ❖ The protection of environment.

The aim of this study is the production of rice husk biosorbent from far north Cameroon to eliminate pollutants from waste water. Specifically we will :

- Product rice husk biosorbent;
- Determine physicochemicals characteristics of biosorbent.

2. Material and Methods

2.1 Material

The material used in the production of biosorbent is essentially constituted by rice husk. They have been collected from non-control site discharge of Yagoua, Department of Mayo Danay, Far North Region of Cameroon (Figure 1). (Latitude 10° 20' 27 N; Longitude 15° 13' 58 E). These waste solids represented in Figure 1 where obtained after the shelling of rice.



Figure 1. Rice husk (Kolwa, Yagoua-Far north Cameroon, march 2018)

2.2 Methods

2.2.1 Preparation of Biosorbent

Rice husk have been soaked in distilled water for about 4 hours and washed in other to eliminate impurities and suspension particles. Then they have been oven dried at 50°C for 24 hours then immersed in phosphoric acid 1M for 24 hours. The mixing has been filtered and washed with distilled water until neutral pH. The residual obtained has been oven dried at 105°C until constant weights. Finally, active rice husk has been crushed and sifted in other to obtain a granulometry less than 500 μm . The biosorbent obtained has been conserved in closed jar. The figure 2 presents the process of the production of rice husk biosorbent.

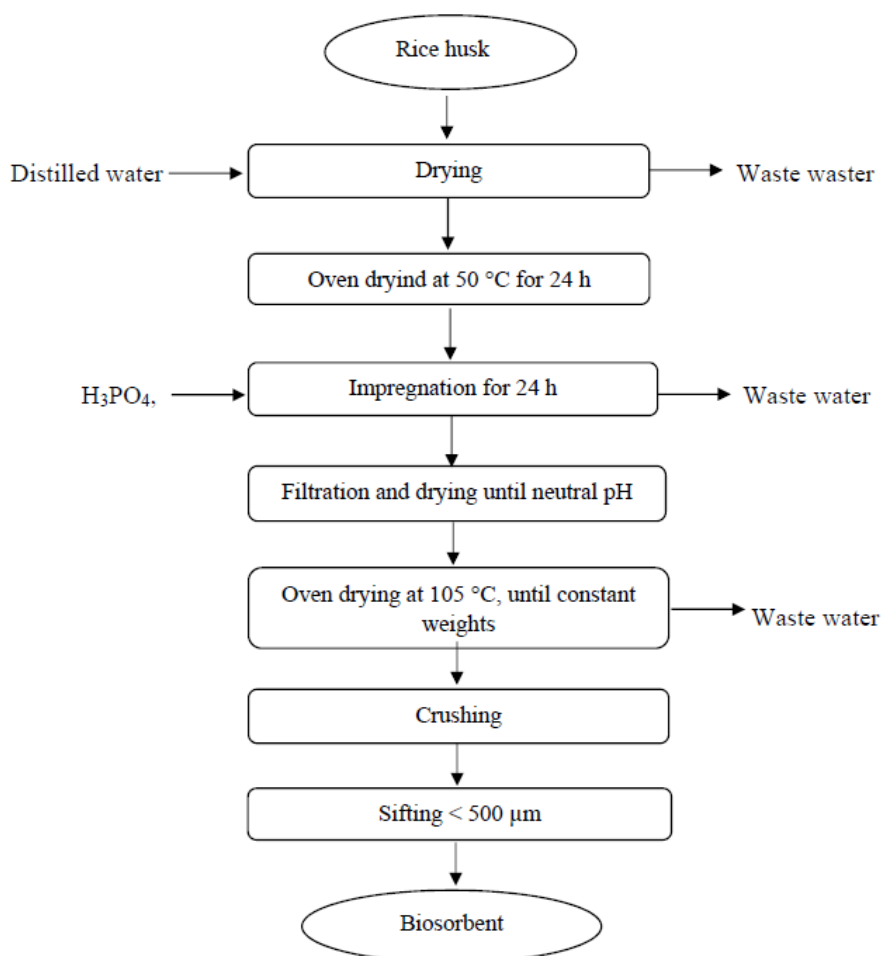


Figure 2. Process of the production rice husk biosorbent

2.2.2 Characterization of Biosorbent

2.2.2.1 Determination of Zero Point Charge (pH_{pzc})

The point of zero charge (pH_{pzc}) is the pH which total charges at solid surface is null. The method describes by Faria *et al.*, (2004) has been used to determine the pH_{pzc} of biosorbent products. This method consists to measure the variation of pH when a solution of chlorhydric acid (0,01M) or soda (0,01M), to 50 ml of a solution NaCl (0,01M) container 0,05g of biosorbent at 25°C. When the pH of NaCl solution is set, we add 0,05g of biosorbent. The hole is shaking for 24 h, and the final pH is written down. We have represented $\Delta\text{pH} = f(\text{pHi})$ where $\Delta\text{pH} = (\text{pHf}-\text{pHi})$. The intersection point between the curve and the straight line $x=0$ corresponding to pH_{pzc} of biosorbent.

2.2.2.2 Determination of Specific Surface by Blue Methylene Method

The principle of the method consists to determine the adsorption capacity of biosorbent by measuring the quantity of Blue Methylene (BM) necessary to cover total surface, of all clayey particules presents in the solution by a monolayer of blue methylene.

The mass of 1g of biosorbent has been mixed in 100 ml beaker with 20 ml of distilled water and shook for 5 minutes at 700 trs/min by magnetic agitator. After, we add with gradual burette, 5 ml of Blue Methylene (10 g/L), the mixture is shaken at 400 trs/min during one minute. With the glass stick, we take one drop of solution that we filter with wattman paper. The test is «positive» if on filter paper, we have a blue central drop surrounded by a blue humid zone. The test is « negative » if the blue central drop is surrounded by an incolore humid zone (AFNOR, 1996). If the test is « positive », we add every one minute, 1 ml of BM for about 5 times to confirm the result. But if the test is « negative », we add 5 ml of BM until we obtain the «positive » test. After obtaining the « positive » test, we note the volume of BM corresponding and we determine the value of the Blue Methylene of biosorbent (VBS) with the formula:

$$\text{VBS} = \frac{v}{m} \times 100 \quad (1)$$

Where v is the volume of BM adsorbed (ml) corresponding to the « positive » test; m the mass of biosorbent. To determine the value of specific surface (SS) we use the formula:

$$\text{SS} = \text{VBS} \times 20,93 \quad (2)$$

2.3 Evaluation of Porosity

Iodine number is an indicator of biosorbent porosity because it measures the porosity of biosorbent. Iodine number is defined as a number of milligramme of iodine adsorped by 1 g of adsorbent (Calvet, 1980). In fact, in erlenmeyer 50 ml, we introduce 0.05 g of biosorbent and 10 ml of iodine solution 0.1 N. We add to the mixture, 2 drops of starch emporium as color indicator. We add drop by drop from burette, 0.1N thiosulfate sodium solution (Faouzia, 2010). The iodine number is determined by formula:

$$\text{Id} = \frac{(V_b - V_s).N.1.5M_I}{m} \quad (3)$$

Where : (V_b-V_s) is the difference between results of test without biosorbent and the test with biosorbent (ml of sodium thiosulfate 0,1N); N , normality of sodium thiosulfate solution (mol/L); M_I , Molar mass of iodine (126,9g/mol); m , the mass of biosorbent (g).

2.4 Fourier Transform Infrared Spectroscopy (FTIR)

Infrared spectra were recorded using an IFS 55 Bruker Fourier transform IR spectrometer equipped with an MCT detector (4000 to 500 cm^{-1}) cooled a 77K and in diffuse reflectance (Harrick attachment) mode. The amount of clay was 70 mg dispersed in 370 mg KBr.

3. Results and Discussion

3.1 Production of Biosorbent

The Figure 3 presents the biosorbent produced. Rice husk have the good appearance and granulometry as low cost adsorbent for growing country. The rice husk as pollutants is easy to be transformed like biosorbent to clean waste water.



Figure 3. Rice husk biosorbent

3.2 Physico-Chemical Characterization of Biosorbent

3.2.1 Point of Zero Charge of Biosorbent

The point of charge (pH_{PZC}) is the intersection point between the curve and the straight-line $x = 0$. Its value is 8.7.

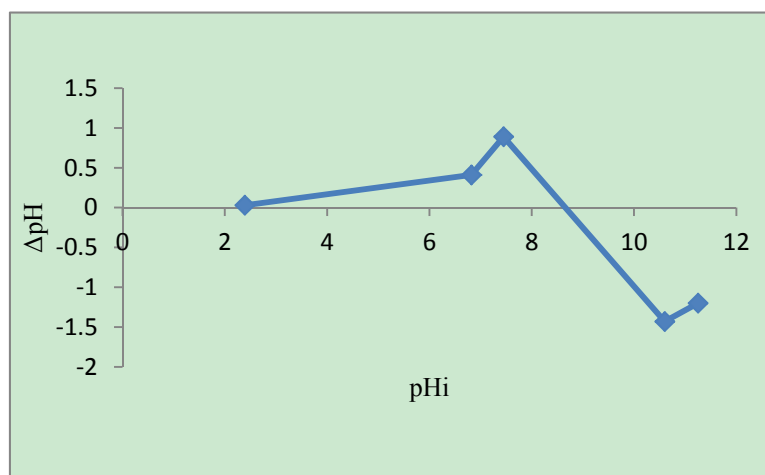


Figure 4. Point of zero charge (pH_{PZC})

The point of zero charge being 8.7, it means that, for a pH less than 8.7, the functions groups on the surface of biosorbent are protonated by protons H^+ excess in the solution; the biosorbent is positively charged and become attractor of adsorbate negatively charged. On the other hand, if the pH of the solution is higher than 8.7, the functions groups on the surface of biosorbent are deprotonated by the presence of OH^- ions in the solution. The biosorbent is negatively charged and become attractor of adsorbate positively charged (Hazourli *et al.*, 2007).

3.2.2 Specific Surface and Iodine Number of Adsorbents

The BM value (VBS) has been determined from BM test. Maximal adsorption is obtained when persistent clear blue ring appear in the periphery of the stain (Figure 5).

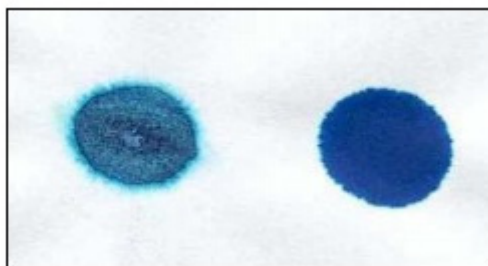


Figure 5. Blue methylene test, left: Positive test and right: Negative test

The surface developed per unit of biosorbent mass and iodine number have been determined. The table 1 presents the result.

Table 1. Specific surface and iodine number of biosorbent

Biosorbent	Specific surface (m^2/g)	Iodine number (mg/g)
	104.65 ± 1	1560.87 ± 1

The table 1 indicates that, the specific surface of biosorbent is $104.65 \pm 1 \text{ m}^2/\text{g}$. According to (Dada *et al.*, 2012), the specific surface of untreated rice husk is $19.8 \text{ m}^2/\text{g}$. So, specific surface of activated rice husk is 5 times those of untreated rice husk. It means that, acid treatment leads the destruction of organic matter (cellulose fiber and lignine granule) of rice husk (Hazourli *et al.*, 2007; Faria *et al.*, 2004), this situation confers to biosorbent a higher specific surface, more and more porous structure. This porous structure favors good adsorption capacity of biosorbent. The analyze of iodine number indicates that it is $1560,87 \pm 1 \text{ mg}/\text{g}$; According to (Hazourli *et al.*, 2007; Sun et Jiang, 2016), the iodine number being higher than $475 \pm 1 \text{ mg}/\text{g}$, so the biosorbent surface is constituted in majority of micropores and can be used for the treatment of waste water.

3.2.3 FTIR

The chemical structure of the adsorbent is of vital importance in understanding the adsorption process. The FTIR technique is an important tool to identify the characteristic functional groups, which are instrumental in adsorption of pollutants. The FTIR spectra of the raw rice husk before sorption were used to determine the vibrational frequency changes in the functional groups in the adsorbent (figure 6).

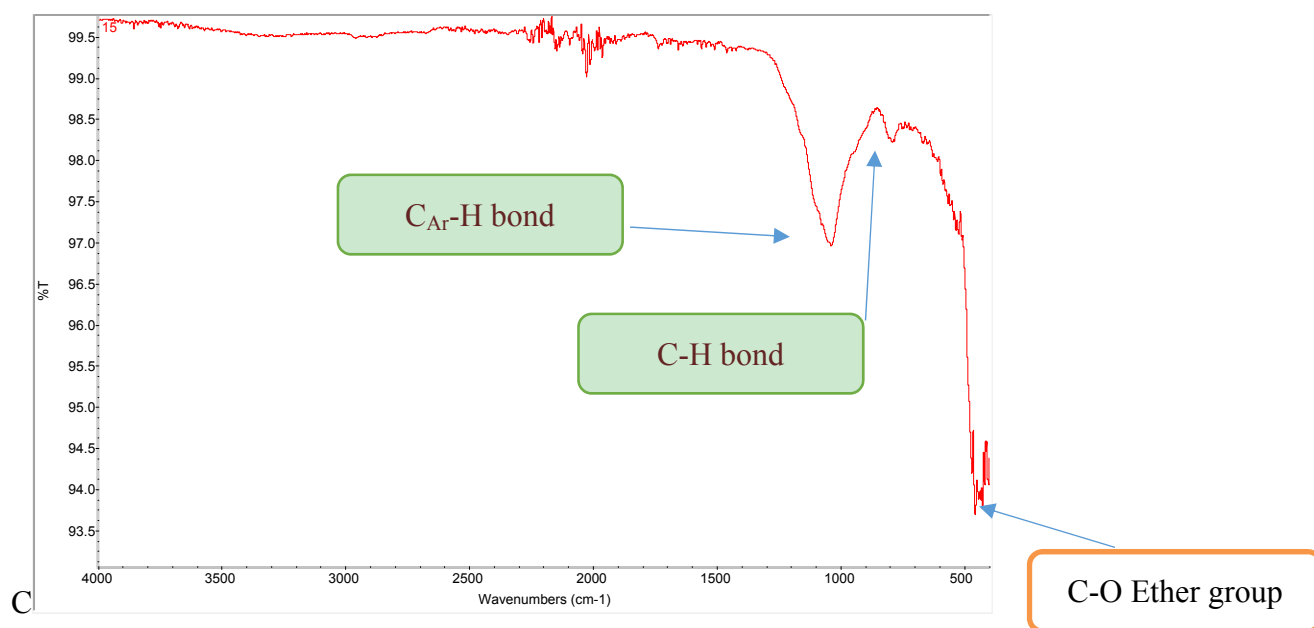


Figure 6. Infrared spectrum

The key chemical groups or bonds of the biosorbent detected by Fourier translation spectrogram were listed in table 2. It was obvious to show that, C-H bond, C_{Ar}-H, C-O (Ether group), was the main chemical group of the biosorbent. Accordingly, one of the most important factor that distinguish the rice husk as adsorbent for wastewater treatment among researchers is the chemical properties (Safa and Bhatti,2011; Chakraborty et al., 2011) that table 2 has showed. The effects of the presence of the surface functional group on adsorption of phenol were analyzed by observing the shifting of the FTIR peaks after the adsorption experiment. Analysis of FTIR shows that the -OH, C-H,-CO, C-OH, Si-OH and -Si-H groups contribute to the adsorption of pollutants onto the surface of adsorbent. In fact The adsorption capacity was greatly influenced by surface group changes.

Table 2. Chemicals functions groups

Wave number (cm ⁻¹)	Chemicals groups or bonds
403.06 – 466,83	C _{tet} -H bond
519,27	C _{Ar} -H bond
785.61	C-H bond
1035.62	C-O Ether group

4. Conclusion

In this study, we showed that it is possible to produce rice husk biosorbent by phosphoric acid treatment. The specific surface of biosorbent is 104,65±1 m²/g; it is 5 times than those of untreated rice husk. Iodine number being 1560,87±1 mg/g, the biosorbent is constituted in majority of micropores. The acid treatment developed better porosity of biosorbent. This situation favorise a good adsorption. The band obtained in the spectrogram indicate the presence of cellulose, hemicellulose and lignin as major constituents of the rice husk. The physicochemical characteristics of the biosorbent are similar to those of activated carbon. In consequence, the valorisation of rice husk contribute to the depollution of waste water and to safeguard the environment. Finally, the rice husk's pollutants yesterday, is low cost adsorbent today and is favorable adsorber for growing country.

From both economics and environmental perspectives, these experimental results warrant further efforts, perhaps in terms of large scale manufacturing and testing.

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