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Activated carbon from bamboo (*Bambusa vulgaris*) waste using CO_2 as activating agent for adsorption of methylene blue and phenol

Carvão ativado a partir de resíduos de bambu (*Bambusa vulgaris*) utilizando CO_2 como agente ativante para adsorção de azul de metileno e fenol

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Abstract

Bamboo (*Bambusa vulgaris*) wastes were used as raw material for producing activated carbon. The materials were collected and turned into activated carbons by carbonization (500 °C, 1.67 °C.min^{-1}, 60 min) and activation (800 °C, 10 °C.min^{-1}, 60 min) processes with CO_2 (100 mL.min^{-1}). The obtained material (CO_2 AC) was characterized by its yield, elemental analysis, ash content, surface area (S_{BET}), Boehm titration method, scanning electron microscopy and used as adsorbent for removing the methylene blue and phenol contaminants. The Langmuir and Freundlich isotherm models were selected for understanding the adsorption process. CO_2 AC produced showed yield of 21.6%, carbon content of 82.13% and S_{BET} of 856.78 m^2.g^{-1}, presenting rapid removal and high adsorption capacity for methylene blue (298.82 mg.g^{-1}) and phenol (558.29 mg.g^{-1})

Keywords: Physical activation; Adsorbent development; Environmental applications

Resumo

Resíduos de bambu (*Bambusa vulgaris*) foram utilizados como matéria-prima para a produção de carvão ativado (CA). Os materiais foram coletados e transformados em CA mediante processos de carbonização (500°C, 1,67°C min^{-1}, 60 min) e ativação (800°C, 10°C min^{-1}, 60 min) com CO_2 (100 mL min^{-1}). O material obtido (CA CO_2) foi caracterizado pelo seu rendimento, análise elementar, teor de cinzas, área superficial (S_{BET}), método titulométrico de Boehm, microscopia eletrônica de varredura e utilizado como adsorvente para remoção dos contaminantes azul de metileno e fenol. Os modelos de isotermas de Langmuir e de Freundlich foram selecionados para entender o processo de adsorção. O CA CO_2 produzido apresentou rendimento de 21,6%, teor de carbono de 82,13% e S_{BET} de 856,78 m^2 g^{-1}, apresentando uma remoção rápida e elevada capacidade de adsorção para o azul de metileno (298,82 mg g^{-1}) e fenol (558,29 mg g^{-1}).

Palavras-chave: Ativação física; Desenvolvimento de adsorvente; Aplicações ambientais

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Introduction

Bamboo belongs to the family Poaceae and subfamily Bambusoideae, sometimes being included in the family Bambusaceae. Bamboo occurs most frequently in tropical and subtropical regions of Asia, Africa and South America, featuring fast growth, high productivity and small production cycles. Small species may reach their maximum height within 30 days and giant species within 180 days; although some species may grow at a rate of 40 cm per day (HIDALGO LOPEZ, 2003; PEREIRA; BERALDO, 2008; KLEINLEIN et al., 2010).

In Brazil, the most commonly grown species is *Bambusa vulgaris*, belonging to the Grupo Industrial João Santos, that has two production units, ITAPAGÉ S.A. Celulose Papéis e Artefatos that manufactures paper for duplex cardboard boxes, located in Maranhão State and CEPASA S.A. Celulose e Papel de Pernambuco S.A. that produces multiwall bags and is located in Pernambuco State, both in Northeast Region, Brazil (PEREIRA; BERALDO, 2008).

The demand for this bamboo species, associated to its potential, growth rate, and quantity of dry matter makes its use feasible, and as a result there is the production of large amounts of waste. The large volume of waste produced is an existing problem in Brazil. Moreover, its discard is seen as an environmental problem (AMAYA et al., 2007; CHEN; XING; HAN, 2009; FELFLI et al., 2011; SUHARTINI; HIDAYAT; WIJAYA, 2011). The market demand together with environmental and social problems justify the use of this waste for developing products with higher value added such as activated carbon (AC).

AC is a highly porous carbonaceous material, with an elevated internal surface area and functional groups in its surface with an adsorption affinity for various contaminants. The AC is obtained from a controlled pyrolysis of a raw material, where temperatures vary from 400 to 1000°C (RIBEIRO, 2005; BRUM et al., 2008; AVELAR et al., 2010; COUTO et al., 2012; CARVAJAL-BERNAL et al., 2015; HÚMPOLA et al., 2016).

In this context, seeking to contribute with better activated carbons that could be used to serve in water treatment, this article discuss these materials production, using bamboo (*Bambusa vulgaris*) wastes as raw material.

Material and methods

Bamboo (*Bambusa vulgaris*) wastes came from Celulose e Papel de Pernambuco S.A. (CEPASA) company, that belongs to Grupo Industrial João Santos, located in Jaboatão dos Guararapes, Pernambuco state, Brazil. Residues were collected, classified in sieves and placed in plastic bags in a climate room at a temperature of 20 ± 2 °C and humidity of 65 ± 5% until reaching constant weight and average moisture content of 12%.

For carbonization, it was used 25 g dry matter, divided into three porcelain crucibles and then inserted in a pyrolysis reactor, starting the carbonization process. The material was heated from 25 to 40 °C, with a heating rate of 1.67 $°C.min^{-1}$ remaining 30 minutes at this temperature. Then the reactor went from 40 to 500 °C with the same heating rate, with a residence time of 60 minutes at the final temperature. Gravimetric yield of charcoal (GY_C) was determined according with Eq. (1).

$$GY_C(\%) = \frac{m_{\text{charcoal}}}{m_{\text{biomass}}} x100 \quad (1)$$

Where: $m_{charcoal}$ = final dried mass (g) of charcoal; $m_{biomass}$ = initial dried mass (g) of the raw material.

The carbonized material was activated in an electric tube furnace, using carbon dioxide (CO_2) as activating agent. The material was inserted into the oven and the ends were closed with pierced rubber stoppers, allowing CO_2 flow. Gas flow was regulated with the use of a rotameter.

The activation process of AC occurred at the final temperature of 800 °C with a heating rate of 10 $°C.min^{-1}$, residence time of 60 min at the final temperature and CO_2 flow was 100 $mL.min^{-1}$ for all activations. The gravimetric yield in activated carbon (GY_{AC}) produced was calculated according with Eq. (2) and the activated carbon was named CO_2 AC.

$$GY_{AC}(\%) = \frac{m_{AC}}{m_{\text{biomass}}} x100 \quad (2)$$

where: m_{AC} = final dried mass (g) of CO_2 AC; $m_{biomass}$ = initial dried mass (g) of the raw material.

Quantification of carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) contents of the raw material and AC produced, approximately 3 mg of sample in duplicate were performed using an elementary simultaneous analyzer. The oxygen content (O), was determined by the Eq. (3), or by difference from the other elements and subtracting the ash content (AsC) present in the raw material.

$$O\% = 100 - C\% - H\% - N\% - S\% - AsC\% \quad (3)$$

For evaluating the ash content, about 1 g of the CO_2 AC was put in a crucible that was taken to a kiln at 103 ± 2 °C, where it stood for 1 hour and 30 minutes. The sample was transferred to a desiccator for cooling. The ash content (AsC) was determined by the material combustion at 750 °C for 6 hours, in a muffle oven, according to Brazilian Regulatory Standard – NBR 8112, Brazilian Technical Norms Association - ABNT (1986).

A porosimeter (ASAP 2020 model from Micromeritics) was used to determine the BET surface area (S_{BET}) - based on N_2 at 77 K (SKAAR, 1988) - the microporous area (S_M), the Langmuir surface area ($S_{Langmuir}$), the external surface ($S_{external}$), the total pore (V_P), the micropore (V_{MP}) and mesopore (V_{MSP}) volumes and the average pore diameter (D) of the CO_2 AC. The sample was degasified at 250 °C.

The determination of the functional surface groups followed the Boehm method (1994). In 0.25 g of the CO_2 AC, 10 mL of NaOH, Na_2CO_3, $NaHCO_3$ (all at 0.05 $mol.L^{-1}$) were added and the material was maintained at agitation at 25°C for 24 hours. After the agitation, the material was filtered in a filter paper (80 $g.m^{-2}$ gramature, 205 μm thickness, 14 μm pores) and 5 mL rates were taken. In the NaOH and $NaHCO_3$ rates 10 mL of HCl (0.05 $mol.L^{-1}$) and in Na_2CO_3 15 mL of HCl (0.05 $mol.L^{-1}$) were added. Next, they were titrated with NaOH (0.05 $mol.L^{-1}$) and the NaOH and HCL standardize. The number of acid groups was determined by considering that sodium hydroxide (NaOH) neutralizes carboxylic acids, lactones, and phenols; wherein sodium carbonate (Na_2CO_3) acts on carboxylic acids and lactones, while sodium bicarbonate ($NaHCO_3$) only neutralizes carboxylic acids.

Scanning electron microscopy - SEM, using 25 k_V, was used to obtain the surface morphology of AC. Samples were mounted on aluminum platform using a double carbon tape with a thin gold layer on an evaporator.

In order to evaluate the application of CO_2 AC in the treatment of contaminants in aqueous medium, adsorption tests were performed using organic pollutants: Methylene blue - MB (cationic dye) and phenol (residue from steel, petrochemistry and pulp and paper industries). Therefore, it was evaluated the kinetics and the adsorption isotherms, using also the models of Langmuir and Freundlich.

In the study of adsorption kinetics, it was used 10 mg of AC and 10 mL of MB and phenol solutions at a concentration of 50 mgL^{-1}. At predetermined intervals of 0.5, 1, 2, 3, 6, 12 and 24 hours, aliquots of solutions containing approximately 3.5 mL were collected and their concentrations were determined. For determining the MB concentrations, it was used wavelength of λ= 665 nm and λ = 270 nm to phenol.

Adsorption isotherms were obtained employing 10 mg of adsorbent and 10 mL of solutions with different concentrations of adsorbates (MB and phenol), which were placed in 20 mL vials and kept stirring at 100 rpm in a table Swivel for 24 hours at room temperature (25 ± 2 °C). Calibration curves were prepared with solutions having concentrations of 25, 50, 100, 250, 500 and 1000 $mg.L^{-1}$ for both MB and phenol. The amount of adsorbate adsorbed per gram of adsorbent (q_{eq}) was calculated in accordance with Eq. (4).

$$q_{eq} = \frac{(C_0 - C_{eq})xV}{m} \tag{4}$$

where: C_0 and C_{eq} = represent, respectively, the initial and at balance concentrations ($mg.L^{-1}$); V = the adsorbate volume (L); and m = the adsorbent mass (g).

The obtained data in the isotherms (q_{eq} and C_{eq}) were adjusted to Langmuir and Freundlich models. Eq. (5) describes the behavior of the Langmuir isotherm, which its linear shape is in Eq. (6), while Eq. (7) is the form of the Freundlich model, which is conveniently linearized by applying logarithm in both terms, see Eq. (8).

$$q_{eq} = \frac{q_m x K_L x C_{eq}}{1 + K_L + C_{eq}} \tag{5}$$

$$\frac{C_{eq}}{q_{eq}} = \frac{1}{q_m x K_L} + \frac{1}{q_m} x C_{eq} \tag{6}$$

where: q_{eq} = adsorbed quantity ($mg.g^{-1}$) for a given adsorbate concentration (MB and phenol); q_m = maximum adsorption capacity ($mg.g^{-1}$); C_{eq} = adsorbate concentration after the equilibrium to be achieved ($mg.L^{-1}$); and K_L = Langmuir constant.

$$q_{eq} = K_F x C_{eq}^{1/n} \tag{7}$$

$$\log q_{eq} = \log K_F + \frac{1}{n} \log C_{eq} \tag{8}$$

where: K_F = sorption coefficient and 1/n = sorption intensity measure (Freundlich constants).

Results and discussion

Gravimetric yield in the charcoal (GY_C) was 35.83% and in the activated carbon (GY_{AC}) was 21.6%. In the mean values for elemental composition of the raw material and of the activated carbon with CO_2 (CO_2 AC) it appears that the raw material has high carbon content, which is desired for AC production (Table 1).

The CO_2 AC had the C content increased and the O and H content decreased. These results are related to the increased release of volatile compounds during the carbonization and activation processes. In addition, there is an increase in C/H ratio and a decrease in O/C ratio. The C/H increase indicates an increase in condensation and flavoring reactions (BRUM et al., 2008; AVELAR et al., 2010; PEREIRA, 2010; COUTO et al., 2012).

It is possible to see that the AC physically produced with CO_2 showed higher N_2 adsorption capacity at low relative pressures (microporous nature). It is noteworthy that the adsorption and desorption isotherms had hysteresis, revealing the presence of mesopores in its structure (Figure 1).

Furthermore, the material has BET surface area of 856.78 $m^2.g^{-1}$; micropores of 716.07 $m^2.g^{-1}$; and external of 140.71 $m^2.g^{-1}$; total pore volume of 0.45 $cm^3.g^{-1}$; total micropores volume of 0.33 $cm^3.g^{-1}$; and average diameter of 21.14 Å.

Table 1 – Elemental composition of raw material and CO_2 AC produced.

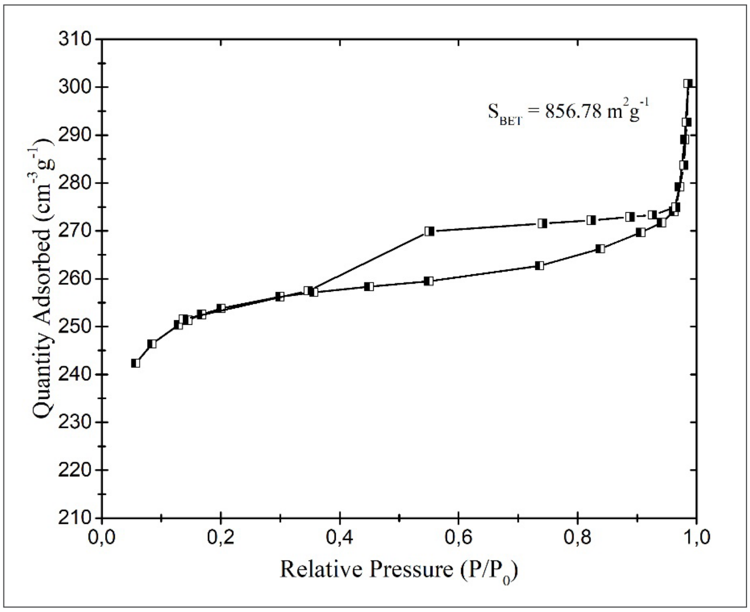
Tabela 1 – Composição elementar da matéria-prima e do CA CO_2 produzido.

Elemental composition	Raw material (%)	CO_2 AC (%)
Carbon (C)	45.25	82.13
Hydrogen (H)	6.13	1.62
Oxygen (O)	44.92	10.19
Nitrogen	1.73	2.34
Sulfur	0.02	0.11
AsC content	1.95	3.61
C/H ratio	7.38	50.70
O/C ratio	0.99	0.12

Source: The authors (2019).

Figure 1 – Adsorption/desorption isotherms of nitrogen (N_2), 77 K, for the physically AC with CO_2

Figura 1 – Isotermas de adsorção/dessorção de nitrogênio (N_2), a 77 K, para o CA fisicamente com CO_2



Source: The authors (2019).

It can be noted that the CO_2 AC presented a relatively high surface area when compared to the S_{BET} values in the literature, which varies from 409 to 658 $m^2.g^{-1}$ (Table 2)

Table 2 – Surface area (S_{BET}) obtained for other adsorbents

Tabela 2 – Área superficial (S_{BET}) obtida para outros adsorventes

Raw material	Activating agent	S_{BET} ($m^2.g^{-1}$)	Reference
Bamboo waste	CO_2	856.78	This work
Babassu nut	CO_2	809.00	Vilella *et al.* (2017)
Apuleia leiocarpa	CO_2	564.90	Nobre *et al.* (2015)
Eucalyptus sp	CO_2	528.00	Couto *et al.* (2012)
Piassava	CO_2	475.00	Avelar *et al.* (2010)
Candeia	CO_2	409.00	Borges *et al.* (2016)

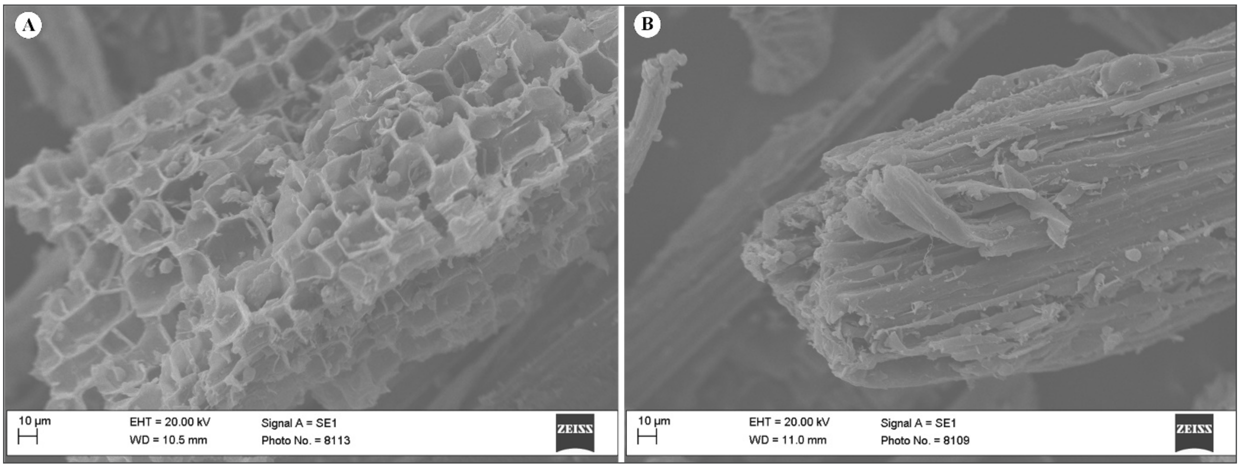
Source: The authors (2019).

AC physically produced with CO_2 from bamboo waste showed acidic functional groups on its surface, with a total acidity of 1.07 mmol.g-1. In terms of larger amounts carboxylic acids stood out with 0.93 $mmol.g^{-1}$, followed by lactone groups with 0.06 $mmol.g^{-1}$ and phenolic with 0.08 $mmol.g^{-1}$.

SEM analysis showed the porous surface in the CO_2 AC formed by the water removal and the volatile components present in the raw material (Figure 2). It is possible to also identify the presence of many open pores in the CO_2 AC, which are due to the activation, which promoted an enlargement of the surface pores allowing rapid diffusion of contaminants (Figure 2A). The activation correlated to the chosen process variables (final temperature, residence time and activating agent) induced a surface cleaning in the CO_2 AC (Figure 2B).

Figure 2 – Surface morphology of CO_2 AC obtained by scanning electron microscopy, to 25 kv voltage. Superficial pores (A) and surface cleaning of CO2 AC (B).

Figura 2 – Morfologia superficial do CA CO_2 obtida por microscopia eletrônica de varredura, para tensão de 25 kv. Poros superficiais (A) e limpeza na superfície do CA CO2 (B).

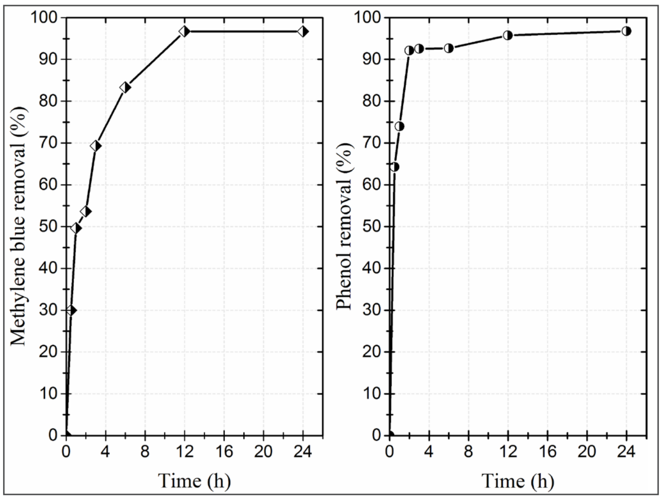


Source: The authors (2019).

For phenol, CO_2 AC showed removal, at 30 minutes, superior to 50%, quickly filling the active sites of the AC. For MB, removal was similar to that observed for phenol (superior to 50%), requiring, however, more time (two hours). Removal of both adsorbates was fast (Figure 3), probably due to the high CO2 AC surface area (856.78 $m^2.g^{-1}$) and to the amount of pore, revealed in the morphology analysis.

Figure 3 – Kinetics of adsorption for MB and phenol to contact time adsorbat/adsorbent (10 mg of AC; 10 mL of 50 $mg.L^{-1}$ solution; pH = 6.8; mechanical agitation of 100 rpm; at room temperature).

Figura 3 – Cinética de adsorção para o azul de metileno - AM e fenol para o tempo de contato adsorbato/adsorvente (10 mg de CA; 10 mL de solução 50 $mg.L^{-1}$; pH = 6.8; agitação mecânica de 100 rpm; em temperatura ambiente).



Source: The authors (2019).

In the values of adsorption isotherms, CO_2 AC was effective in removing both contaminants (Figure 4). In the highest analyzed concentration (1000 mg.L-1), CO_2 AC showed high levels in the adsorbed amount ($q_{eq)}$ for MB (345.22 $mg.g^{-1}$) and phenol (547.72 $mg.g^{-1}$). The highest adsorption capacity for phenol when compared to the MB is associated with larger CO_2 AC micropore area.

In Table 3 the value coefficient (R^2) for adsorption isotherms for MB and phenol were best fit for the Freundlich model, which enables multilayer adsorbate adsorption with weak interactions between adsorbate and adsorbent. The CO_2 AC showed high qm for MB 298.82 $mg.g^{-1}$ and 558.29 $mg.g^{-1}$ for phenol.

Table 3 – Langmuir and Freundlich parameters for MB and phenol compounds.

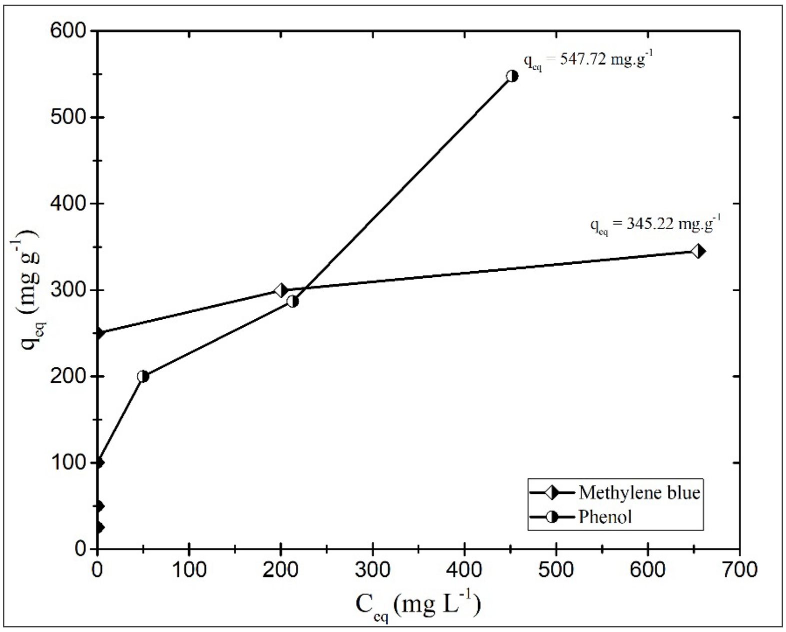
Tabela 3 – Parâmetros de Langmuir e Freundlich para os compostos AM e fenol.

Compounds	**Langmuir**			**Freundlich**		
	q_m ($mg.g^{-1}$)	**K_L ($L.mg^{-1}$)**	**R^2**	**1/n**	**KF [($mg.L^{-1}$)($L.mg^{-1}$)$^{1/n}$]**	**R^2**
Methylene blue	298.82	0.027	0.876	0.28	58.95	0.881
Phenol	558.29	0.008	0.913	0.71	6.84	0.978

Where: q_m = maximum adsorption capacity ($mg.g^{-1}$); K_L = Langmuir constant; R^2 = determination coefficient; 1/n = Freundlich parameter; and K_F = Freundlich constant.

Figure 4 – Adsorption isotherms for MB and phenol by CO_2 AC (24 hours; 10 mg of AC; 10 mL of solutions in different concentrations; q_{eq} = adsorbed quantitate; C_{eq} = equilibrium concentration).

Figura 4 – Isotermas de adsorção para o AM e fenol pelo CA CO_2 (24 horas; 10 mg de CA; 10 mL de soluções em diferentes concentrações; q_{eq} = quantidade adsorvida; C_{eq} = concentração de equilíbrio).



Source: The authors (2019).

Table 4 presents the maximum adsorption capacity (q_m) of CO_2 AC and of other well-known materials for removal of contaminants in the literature. It can be noted that the CO_2 AC presented a high adsorption capacity when compared to the q_m values in the literature, which varies from 32 to 213 $m^2.g^{-1}$.

Table 4 – Comparison of the maximum adsorption capacity (q_m) by other materials

Tabela 4 – Comparação da capacidade máxima de adsorção (q_m) por outros materiais

Raw material	Activating agent	Contaminants	q_m ($mg.g^{-1}$)	Reference
Bamboo	CO_2	Methylene blue	298.82	This work
Bamboo	CO_2	Phenol	558.29	This work
Cocoa	CO_2	Methylene blue	212.77	Ahmad *et al.* (2012)
Piassava	CO_2	Phenol	137.95	Avelar *et al.* (2010)
Candeia	CO_2	Phenol	98.20	Borges *et al.* (2016)
Eucalyptus sp.	CO_2	Methylene blue	32.00	Couto *et al.* (2012)

Source: The authors (2019).

Conclusions

Bambusa vulgaris wastes were good precursors for production of good quality activated carbon. CO_2 AC produced showed GY_{AC} of 21.6%, carbon content of 82.13% and S_{BET} of 856.78 $m^2.g^{-1}$, presenting rapid removal and high adsorption capacity for MB (298.82 $mg.g^{-1}$) and phenol (558.29 $mg.g^{-1}$).

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