

# Production of efficient activated carbons from spent brewery grains for the removal of antibiotics from water- A fractional factorial design

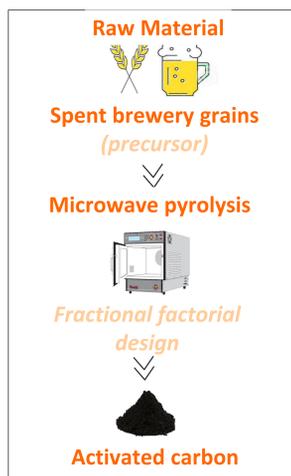
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## Objectives

The aim of this work was to produce an activated carbon (AC) by microwave-assisted pyrolysis of spent brewery grains (SBG), used for removal of the antibiotics sulfamethoxazole (SMX), trimethoprim (TMP) and ciprofloxacin (CIP) from water. A fractional factorial design (FFD) was applied to select the optimum conditions to produce AC from SBG using microwave-assisted pyrolysis.

## Methodology – FFD

The factors that significantly can influence the AC properties, namely, activating agent (AA) (KOH and K<sub>2</sub>CO<sub>3</sub>), AA: precursor ratio (1:1, 1:2 and 1:5), temperature of pyrolysis (600 °C, 700 °C and 800 °C) and residence time (10 min, 20 min and 30 min) were evaluated. Then, the impact of the referred production variables on AC surface area ( $S_{BET}$ ), yield of production (%), total organic carbon and on the adsorptive removal (%) of SMX, TMP and CIP by the produced materials were evaluated (Fig. 1).

## Results

Table 1. Experimental conditions for the produced ACs.

Variables				
AC	Activating agent (AA)	AA: precursor	Temperature (°C)	Residence time (min)
1	KOH	1:1	600	20
2	KOH	1:1	700	20
3	KOH	1:1	800	30
4	KOH	1:2	600	30
5	KOH	1:2	700	20
6	KOH	1:2	800	10
7	KOH	1:5	600	30
8	KOH	1:5	700	10
9	KOH	1:5	800	10
10	K <sub>2</sub> CO <sub>3</sub>	1:1	600	10
11	K <sub>2</sub> CO <sub>3</sub>	1:1	700	10
12	K <sub>2</sub> CO <sub>3</sub>	1:1	800	30
13	K <sub>2</sub> CO <sub>3</sub>	1:2	600	10
14	K <sub>2</sub> CO <sub>3</sub>	1:2	700	30
15	K <sub>2</sub> CO <sub>3</sub>	1:2	800	20
16	K <sub>2</sub> CO <sub>3</sub>	1:5	600	20
17	K <sub>2</sub> CO <sub>3</sub>	1:5	700	30
18	K <sub>2</sub> CO <sub>3</sub>	1:5	800	20

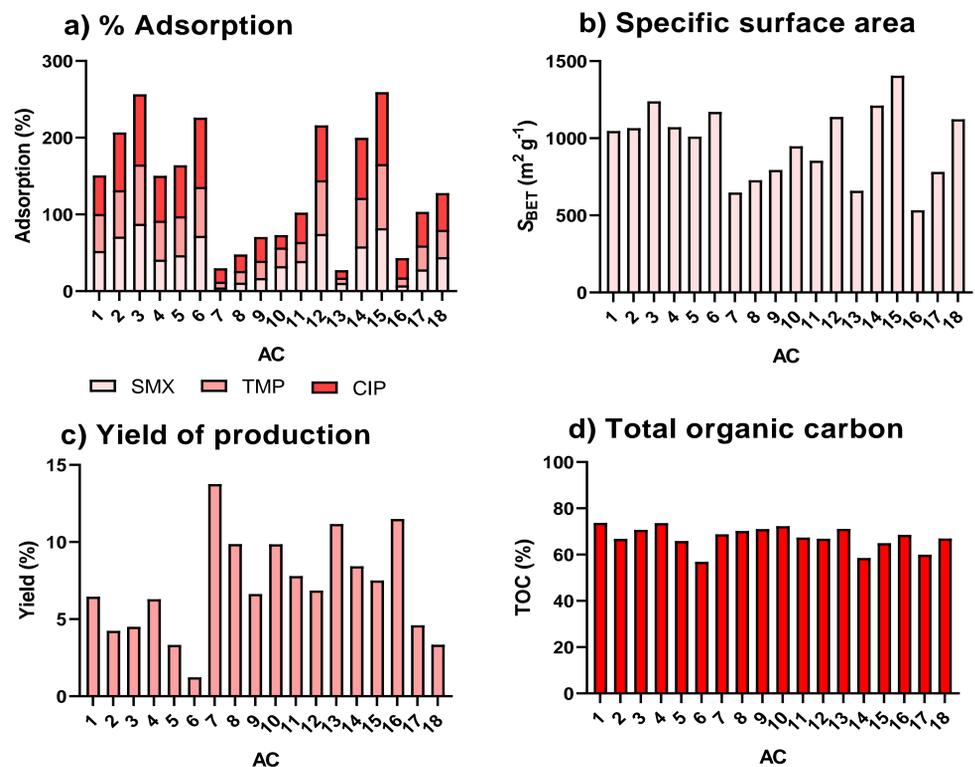


Fig. 1 a) Adsorption percentage of SMX, TMP and CIP, b) surface area, c) yield of production and d) total organic carbon obtained for eighteen AC materials.

Under optimization conditions, a microporous AC (AC15) (see production conditions in Table 1) was selected, with  $S_{BET}$  of  $1405 m^2 g^{-1}$  (Fig. 1 b) and removal percentages of antibiotics from water ranging between 82% to 93%, was obtained and selected for kinetic and equilibrium adsorption studies (Fig. 2 and Fig 3).

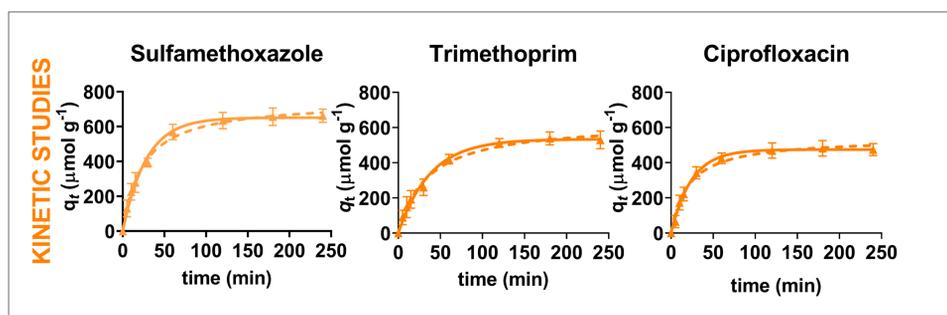


Fig. 2 Kinetic studies of SMX, TMP and CIP onto AC15 from ultrapure water. Pseudo-first order (full line) and pseudo-second order (dashed line). Experimental conditions:  $C_i$  20  $\mu mol L^{-1}$ ; dose 15  $mg L^{-1}$  and 25 °C. Note:  $q_t$  ( $\mu mol g^{-1}$ ) = adsorbed concentration at a time  $t$  (min).

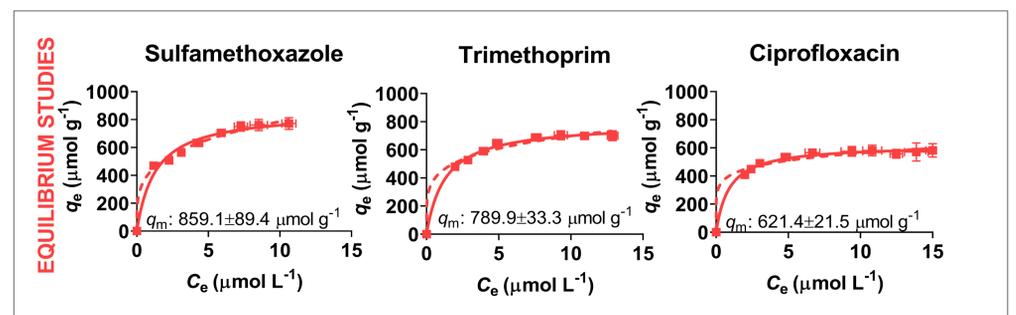


Fig. 3 Equilibrium studies of SMX, TMP and CIP onto AC15 from ultrapure water. Langmuir (full line) and Freundlich (dashed line).  $C_i$  20  $\mu mol L^{-1}$ ; dose 10-50  $mg L^{-1}$  and 25 °C. Note:  $q_e$  ( $\mu mol g^{-1}$ ) = adsorbed concentration at the equilibrium;  $q_m$  ( $\mu mol g^{-1}$ ) = maximum monolayer adsorption capacity; and  $C_e$  ( $\mu mol L^{-1}$ ) = equilibrium concentration in the aqueous phase.

## Conclusions

The procedure developed in this work not only avoided the use of longer pyrolysis times, which can be extended to several hours in conventional furnaces, but also the use of higher proportions of activating agent typically applied in the production of AC. The AC obtained under pyrolysis at 800 °C during 20 min and applying K<sub>2</sub>CO<sub>3</sub>: precursor ratio of 1:2 exhibited an  $S_{BET}$  of  $1405 m^2 g^{-1}$  and very high percentage of removal antibiotics, for a dose of material of only 25  $mg L^{-1}$ .

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