Systematic optimization of the thermal reactivation of activated carbon for water treatment using statistical methods

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Open-Minded

Motivation & Targets

The widespread use of activated carbons (ACs) for various industrial purposes has led to a steady increase in the demand for fresh ACs worldwide. An energy efficient way to tackle this problem is the regeneration of exhausted ACs, whereas thermal regeneration is considered as one of the most efficient regeneration processes. Therefore, this work aims at the systematic optimization of the process parameters influencing the thermal reactivation process of ACs for water purification. In order to assess the reactivation success, the regenerated ACs (RC) should be analyzed for their structural as well as physical-chemical properties and compared with the respective fresh (FC) and loaded ACs (LC). Finally, the software Design-Expert should be used to establish empirical, mechanistic relationships between the influencing variables and the respective quality characteristics, to perform a sensitivity analysis of the individual influencing variables and to determine the optimum reactivation parameters.

Experimental & Methods

Thermal reactivation process

Fig. 1. Reactivation experiments using a rotary kiln

Reactivation protocol:

- Rotary kiln is first heated up to 300°C in each test
- Final temperature at the end of the tests varied between 450°C and 900°C
  - Below 450°C the reactivation success is too low
  - Above 900°C the pore system collapses, or too high mass loss occurs
- No holding plateaus

Design of experiment

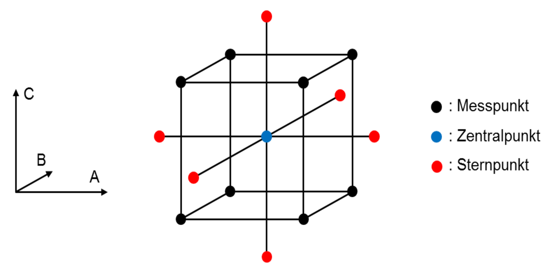

Table 1: Variable factors and their step values

Factors	Step 1	Step 2
Heating rate [°C/h]	225	450
Holding time [min]	40	80
H_2O/N_2 ratio	0	0.3
CO_2/N_2-ratio	0	0.3

Targeted responses:

- Inner surface area (m^2/g) (**R_1**)
- Energy consumption (kW/h) (**R_2**)
- RAC yield (%) (**R_3**)
- Adsorption capacity (m^2/g) (**R_4**)

Structural and chemical characterization

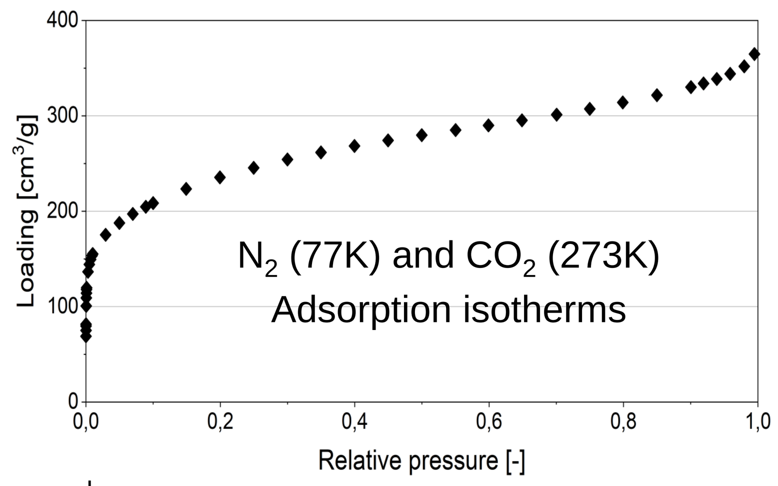

Fig. 2. Adsorption isotherm

Targeted variables:

- Inner Surface Area (BET)
- Pore Volume
- Micropore Volume
- Pore Size Distribution (PSD)

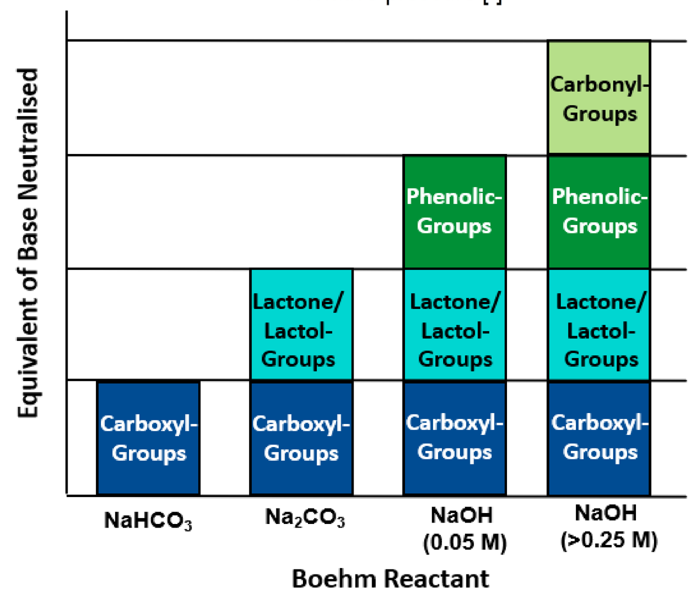

Fig. 3. Boehm bases and assigned surface groups

Boehm titration:

- Chemical method for the quantitative determination of acidic oxidic surface groups on activated carbons
- Selective neutralization of functional groups with bases of different acidity
- Amount of surface groups can be correlated with the respective base consumptions

Results & Discussion

Table 2: Selected results and reactivation conditions of FC, LC and RC

AC	BET area [m²/g]	Total pore volume [cm³/g]	Micropore volume [cm³/g]	Heating rate [°C/h]	Reactivation time [min]	Final Temperature [°C]	H_2O/N_2 ratio	CO_2/N_2 ratio
FC	842.7	0.544	0.299	-	-	-	-	-
LC	419.3	0.313	0.139	-	-	-	-	-
RC1	698.9	0.484	0.244	337.5	60	637.5	0.15	0.15
RC2	690.5	0.445	0.250	337.5	60	637.5	0.15	0.15
RC3	769.1	0.523	0.273	450	80	900	0.3	0.3
RC4	596.8	0.414	0.212	225	40	450	0.3	0
RC5	643.5	0.435	0.227	225	40	450	0.3	0.3
RC6	734.7	0.492	0.261	450	80	900	0	0.3
RC7	699.9	0.472	0.247	337.5	60	637.5	0.15	0.4

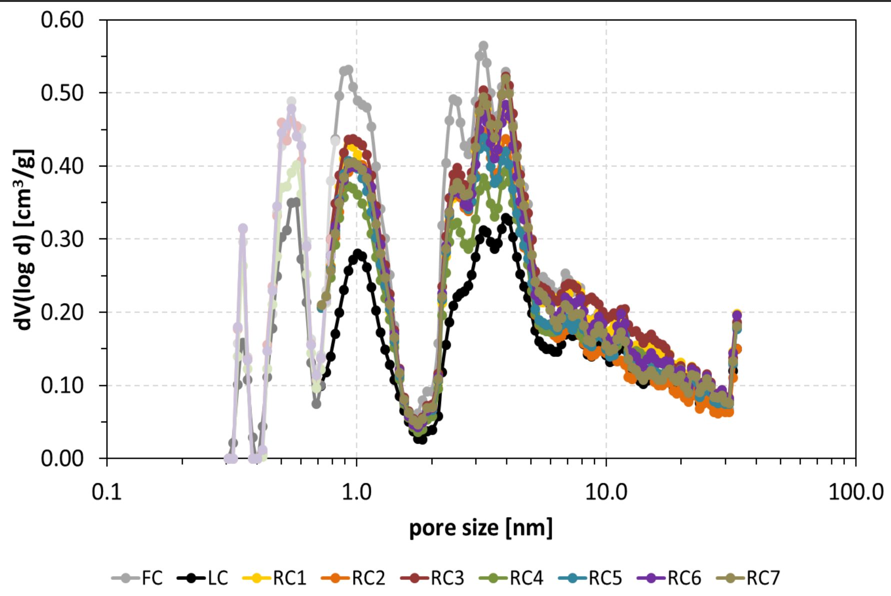

Fig. 4. Pore size distributions from N_2 (dark) and CO_2 (bright) isotherms

- Micro- and mesopores
- Pores blocked in process
- Blocking removed during reactivation (restoration of pore structure)
- No shift in pore size distribution

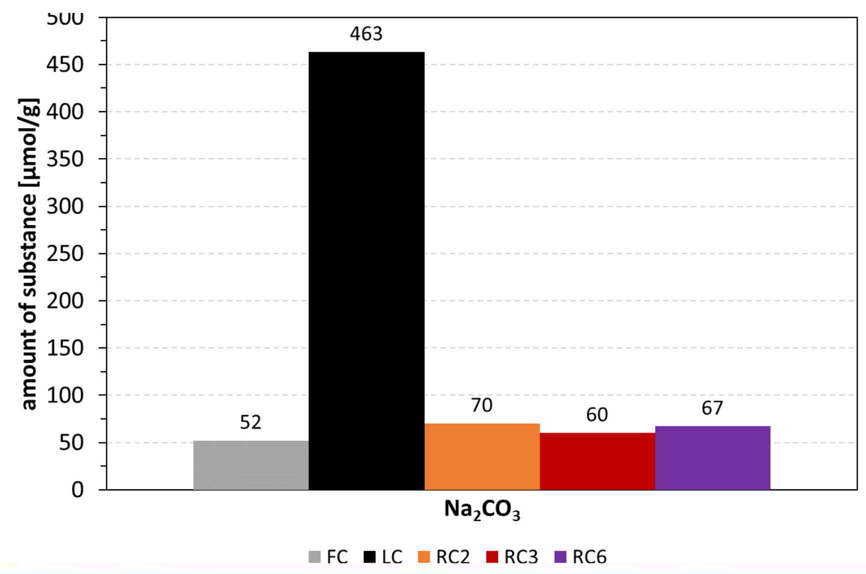

Fig. 5. Total amount of phenol and lactone / lactol groups

- Highest amount of groups for LC
- RC amount of groups close to the FC
- Acidic oxidic surface groups are created in process and removed during reactivation
- No differences between regenerated AC

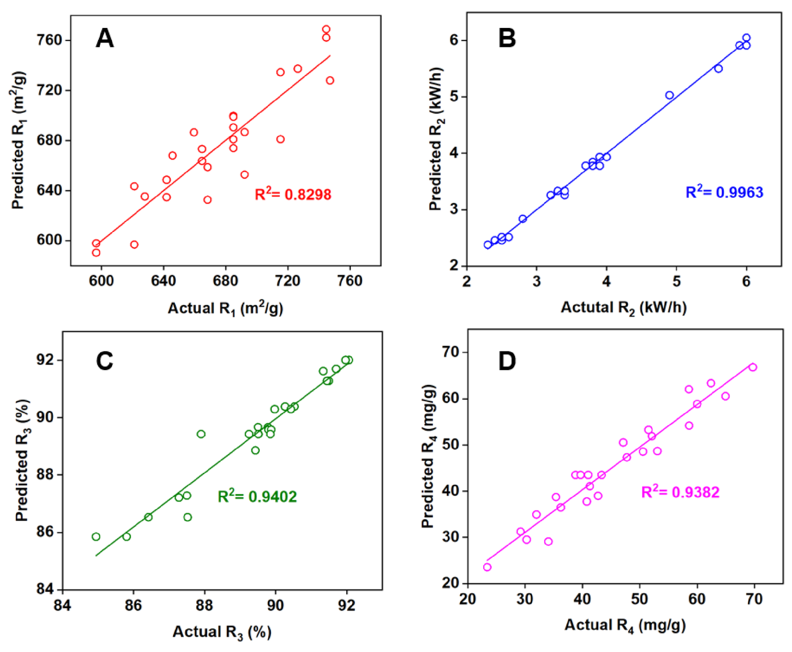

Fig. 6. Evaluation of the developed polynomial model for (A) R_1, (B) R_2, (C) R_3 and (D) R_4

The proximity of R^2 value to 1 implied the adequacy of the developed regression model and showing a qualitative agreement between the actual and predicted values

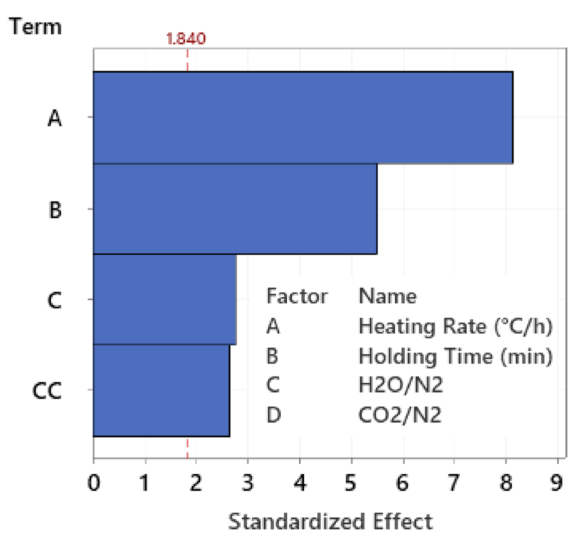

Fig. 7. Pareto chart of the standardized effects for inner surface area

- Inner surface area is significantly influenced by both heating rate and holding time followed by H_2O/N_2 ratio
- CO_2/N_2 ratio showed no effects on surface area

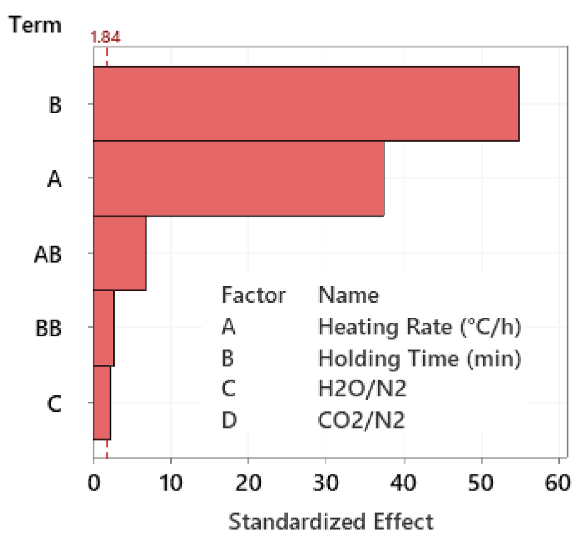

Fig. 8. Pareto chart of the standardized effects for energy consumption

- Energy consumption is greatly influenced by the holding time followed by heating rate
- H_2O/N_2 ratio and CO_2/N_2 ratio showed almost no influence on the energy consumption

Summary & Outlook

- ✓ Restoration of the inner surface area up to 91% of the FC was achieved
- ✓ Pore volume increases with rising inner surface area
- ✓ Higher final temperature leads to larger inner surface area
- ✓ Heating rate and holding time played the most significant roles on all the targeted responses
- ✓ CO_2/N_2 ratio did not show any influence on the targeted responses
- ✓ In line with low cost, low energy and low CO_2 footprint, this study also reduced the dependency on fresh AC and minimized the waste disposal
- ✓ It is anticipated that this study might be helpful for the AC manufactures to adjust their operating parameters of reactivation furnaces

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