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OPTIMIZATION OF THE PRODUCTION OF ACTIVATED CARBON FROM FAST PYROLYSIS CHAR

Introduction

Fast pyrolysis of biomass yields approximately 10-20% biochar by mass, which currently is considered a low-value co-product. Through further activation techniques, biochar can be converted into activated carbon (AC). The yield and quality of AC is a result of several different parameters such as: temperature and pressure, gas type and flow rates, feedstock type and pretreatment, heating rates and holding times, etc.

In order to efficiently investigate activation methods with a large amount of operating conditions other more sophisticated statistical methods such as Response Surface Methodology (RSM) were explored.

Objectives

- Produce Activated carbon from red oak fast pyrolysis biochar.
- Use Response Surface Methodology to optimize production, performance and economics of the process.

Materials and Methods

Feedstock: Red Oak (RO) fast pyrolysis biochar was produced in a fluidized bed reactor (~500°C and ~20s residence time).

Material pretreatment: methanol was used to wash RO at rate of 8:1 v/v (methanol to biochar) to remove remaining bio-oil in the sample. Acid wash with 0.1 M of sulfuric acid followed, at a rate of 10:1 v/v (acid solution:biochar) and dried at 105°C for 48 hrs.

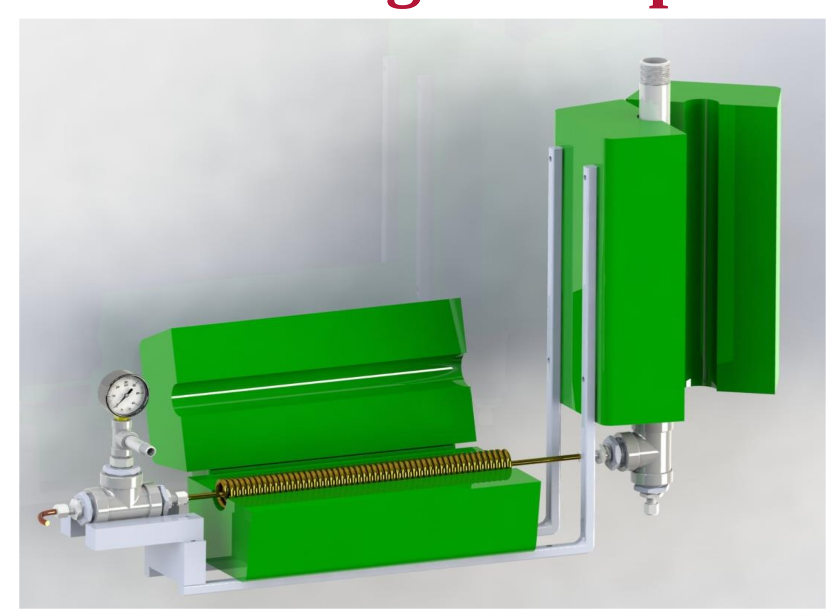
Experimental design: A complete factorial design with 3 temperature levels by three levels of activation times with 3 replications was performed.

Biochar Activation: Roughly 1 gram of biochar was activated in every run. Steam continuously flushed the sample at 1mL/min/gr for the designed temperature and residence time.

Physi-sorption analysis: 0.1g of sample was degassed for 4 hours at 300°C with a vacuum reaching at least 100 Pa. Afterwards, samples were measured for BET surface area. Promising biochar-activated carbons were analyzed for the volume of micropores based on v-t plot, and pore size distribution based on Quenched Solid Functional Theory for disordered carbonaceous materials.

Activated and non activated biochars present a type II isotherm as described by Brunauer, Deming and Teller. Non-activated fast pyrolysis chars present very low surface area typically less than $10 m^2/g$ and mostly representing external surface area.

Reactor design and operation



Operating parameters:
- Temperature: 400-800°C
- Residence time: 5-60min
- Steam flow: 1 mL/g BC/min
- Pressure: 0-3 PSI
- Sample size 1-2 g
- Extra port for adding gases
- 2 Temperature controllers

Figure: Batch reactor used for steam activation

Results and Discussion

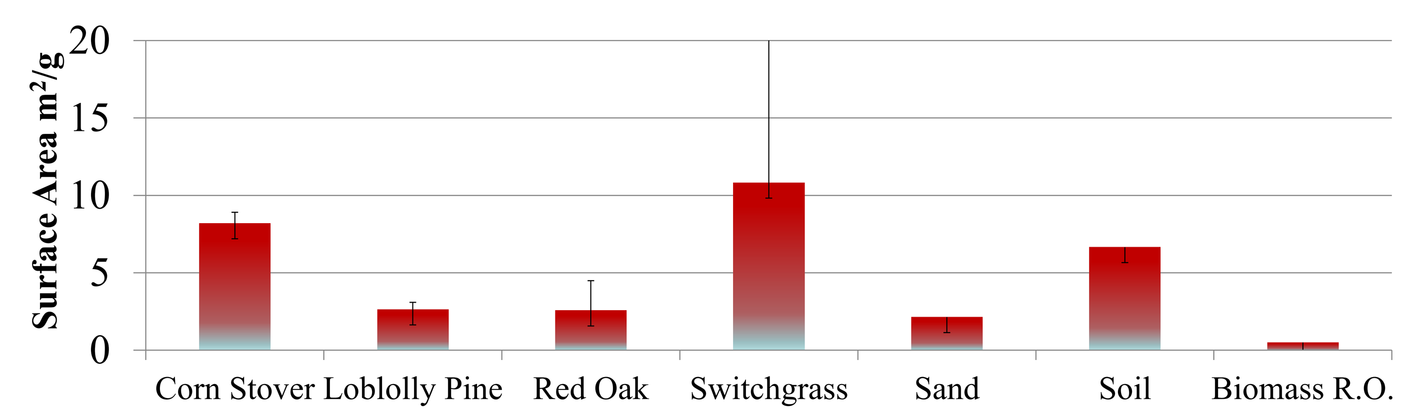


Figure: BET surface area for non activated Fast Pyrolysis Biochar

Note that commercial activated carbon typically range from 500 to 1500 m^2/g

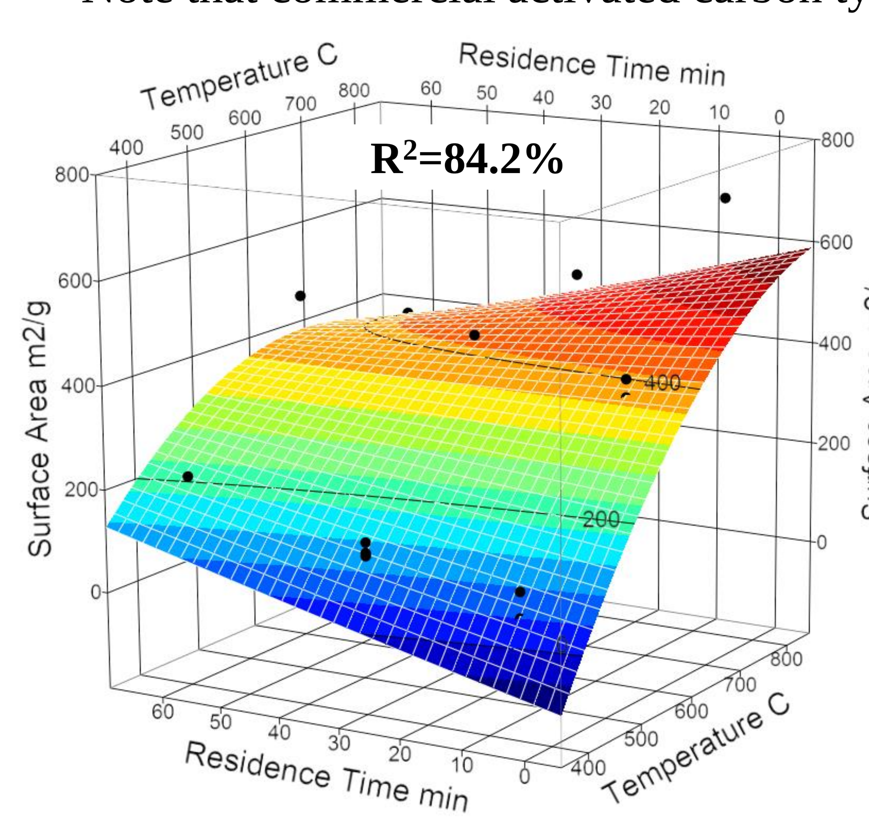
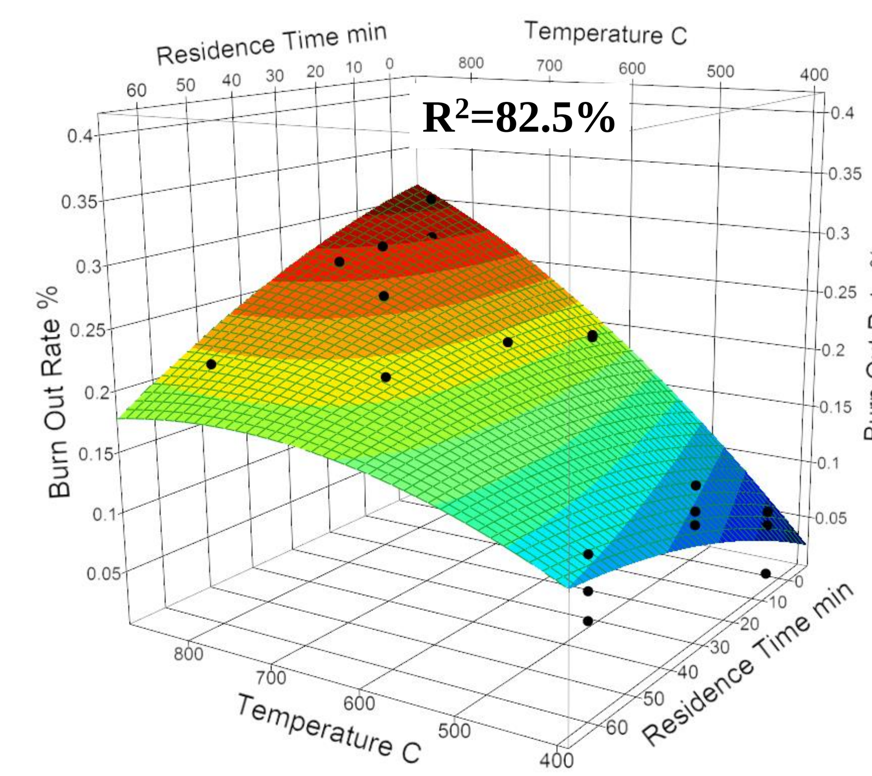


Table: Surface area model; estimated parameters and level of significance

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	370.5	38.1	9.72	<.0001
Temp	176.4	21.7	8.14	<.0001
Res. Time	-6.3	22.9	-0.27	0.788
Temp*R.Time	-69.6	26.3	-2.64	0.018
Temp²	-84.5	38.6	-2.19	0.044
Res. Time²	1.4	36.9	0.04	0.971

Figure: RSM for biochar surface area for different temperatures and holding times.

The predicted model closely fits the experimental data (R^2=84%), with significant quadratic term for temperature and interaction between residence time and temperature. The overall model is significant p< 0.0001 and level of significant for the lack of fit test is p=0.26. The quadratic model is appropriate and predicts very well the experimental data.



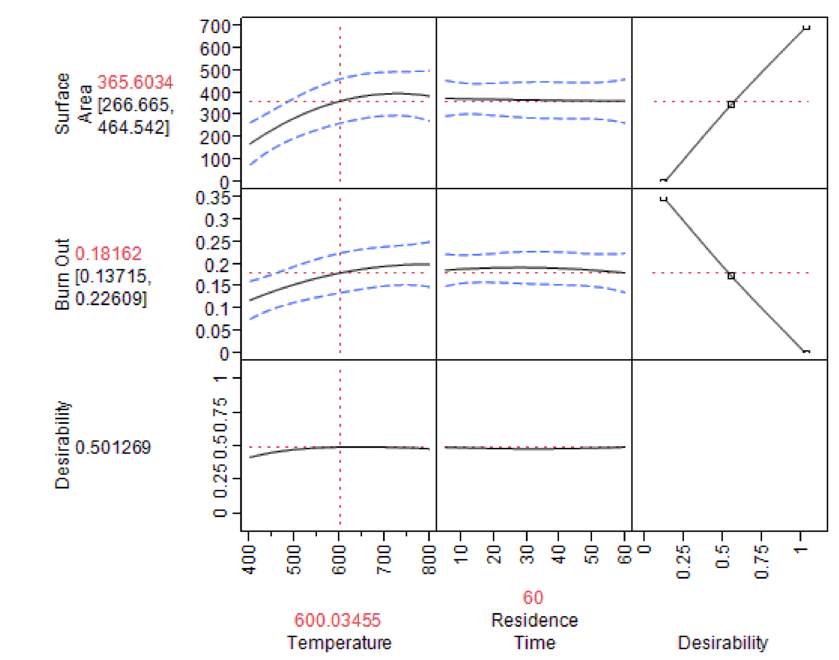


Figure: Predictor profiler and desirability functions for surface area and burn off

Figure: RSM for burn off rate with different temperatures and residence time

The maximum desirability function for surface area. and burn off resulted in 365m2/g and 18%, which was predicted for 600°C and 60 min of residence time.

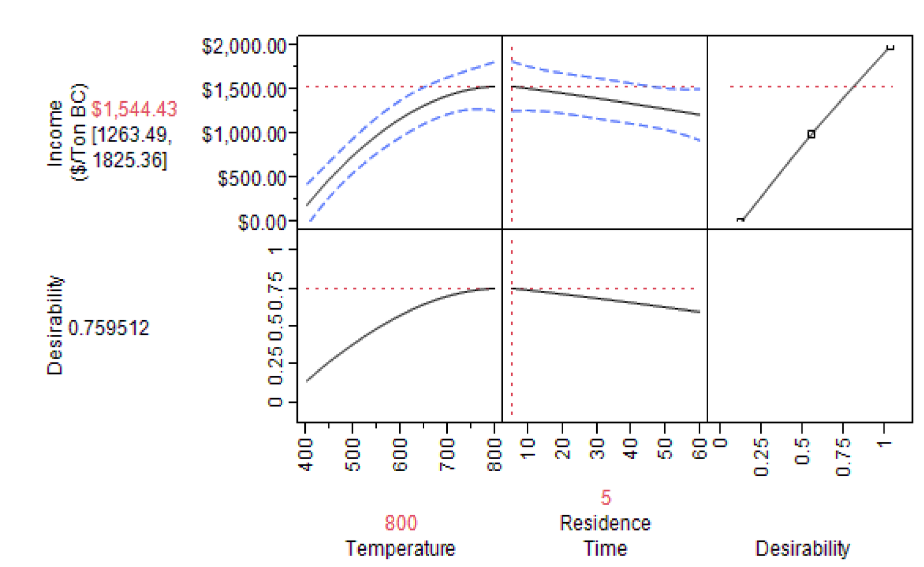


Figure: Predictor profiler and desirability functions for gross income $/ton of biochar

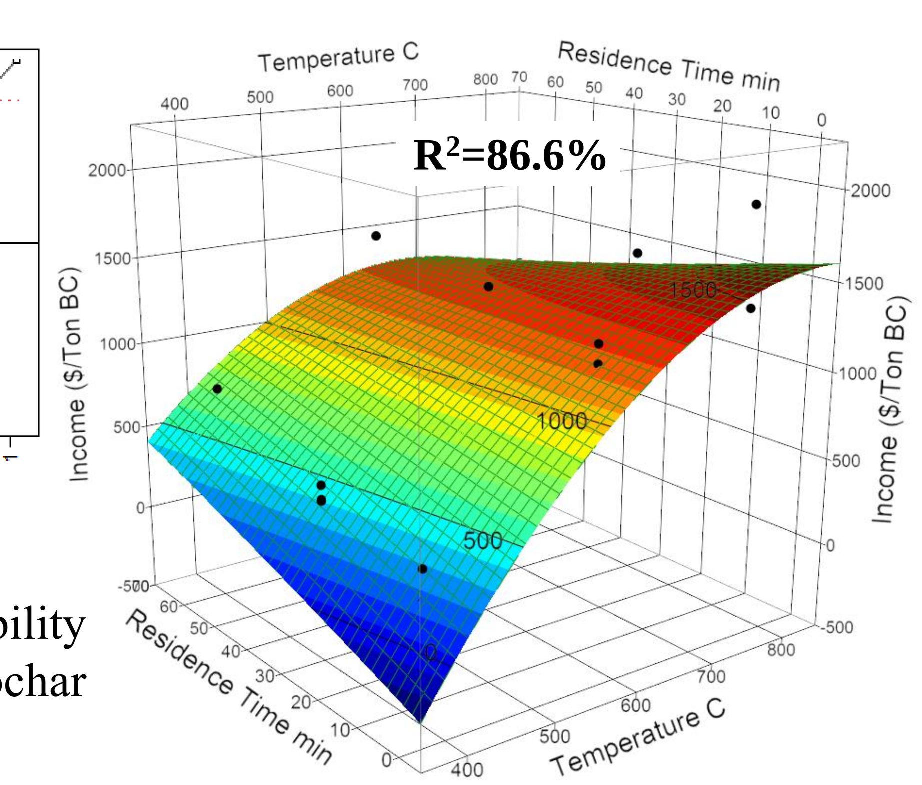


Figure: Gross income $/ton of biochar for the different treatment combinations

Based on hypothetical value, of \$4 per m^2/g for commercial activated carbon, an optimum region can be found for maximizing income. Highest gross income for activating biochar was reached around 800°C and 5 min of residence time. At this condition, approximately \$1540 can be gained for every ton of activated biochar.

Conclusion

- Low cost adsorbents can be produced by steam activation of fast pyrolysis char.
- Red oak chars were converted into activated carbon (>500 m^2/g)
- The use of RSM helped to optimize activation parameters and experimental work (by decreasing runs and replication) and optimizing process economics.

Future Work

- Chemisorption and functional attributes should be studied to identify different uses and applications of activated biochars.

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