Paper Number: 04

Effect of Activation Agent's Impregnation Route on Activated Carbon Properties

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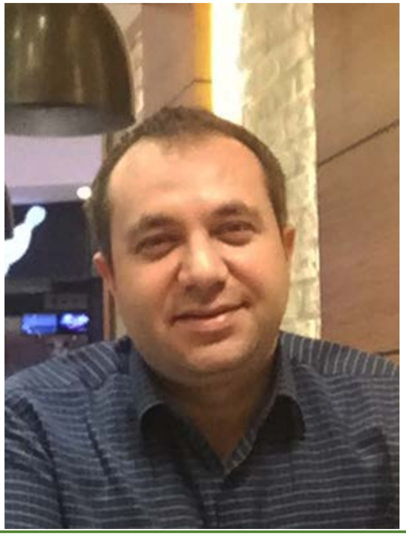

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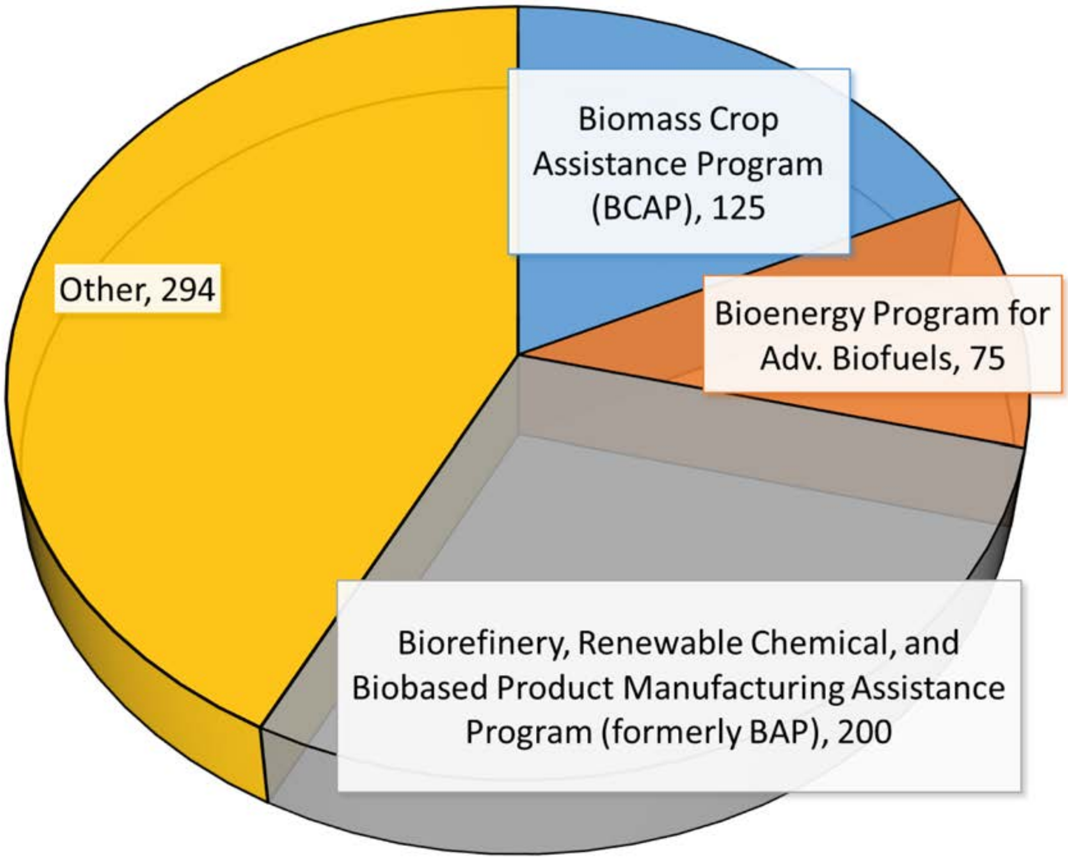
Biomass Crops and the United States



Total Mandatory Funding Authorized= $694 Million (2014 Farm bill)

Total Discretionary Funding Authorized= $765 Million

Bracmort, K. Energy Provisions in the 2014 Farm Bill (P.L. 113-79): Status and Funding. Congressional Research Service document# R43416. Available at: https://fas.org/sgp/crs/misc/R43416.pdf

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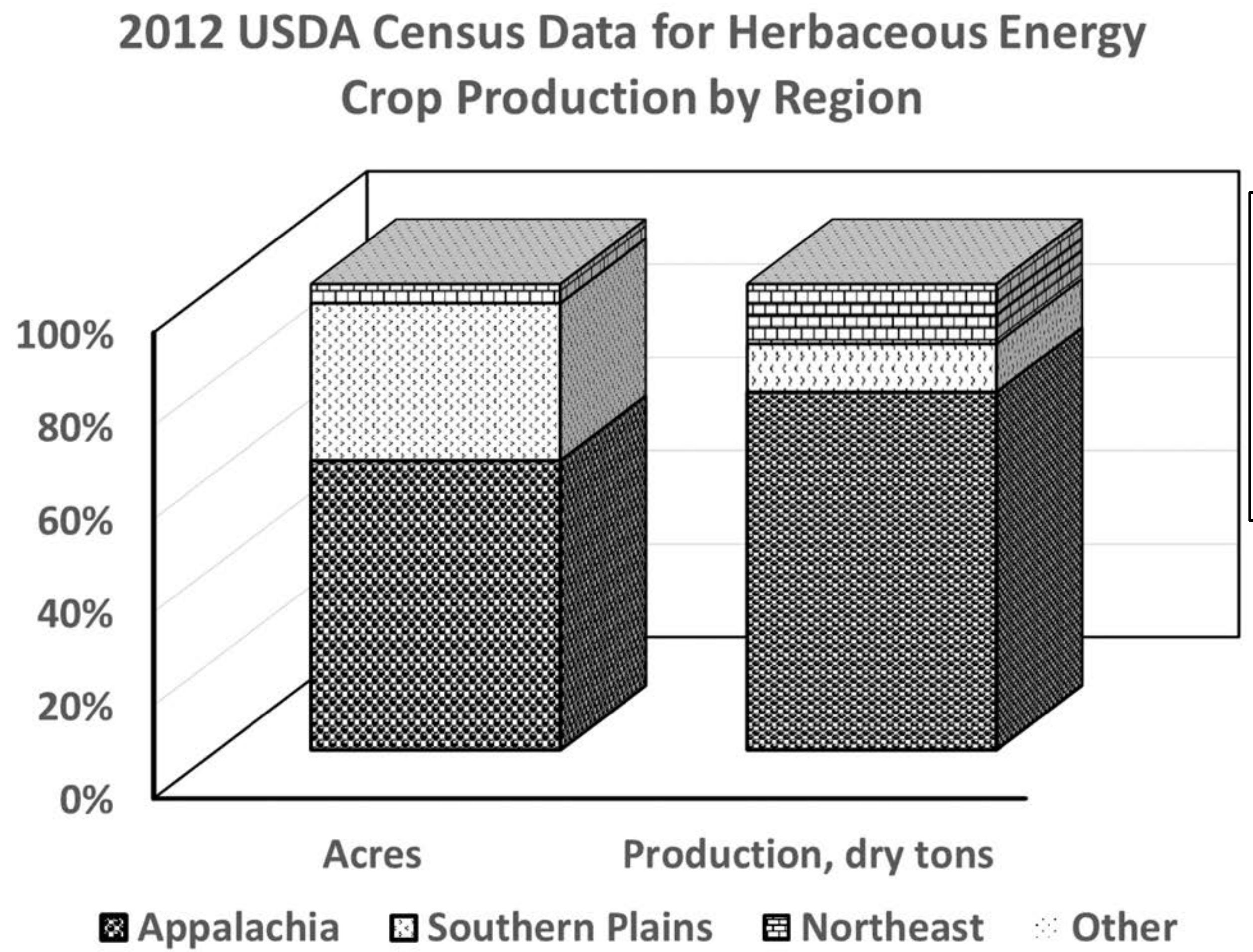

Total Investments by the United States Government= ????

Total Acreage- 2,889 acres
Total Harvest- 11,224 dry tons
Total Consumption for Energy and Energy Products- 0

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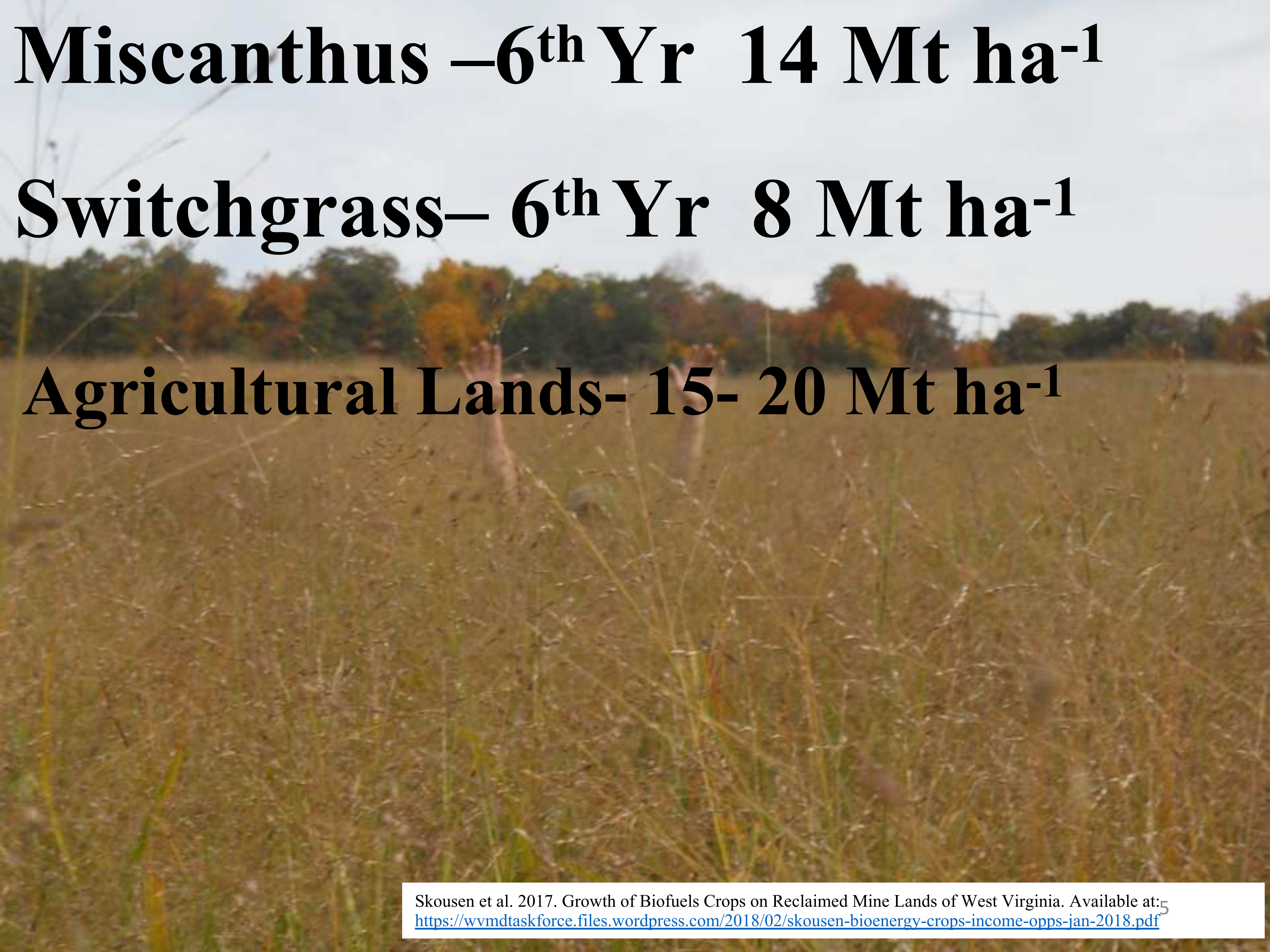
U.S. Department of Energy. 2016. *2016 Billion-Ton Report: Advancing Domestic Resources for a Thriving Bioeconomy, Volume 1: Economic Availability of Feedstocks*. M. H. Langholtz, B. J. Stokes, and L. M. Eaton (Leads), ORNL/TM-2016/160. Oak Ridge National Laboratory, Oak Ridge, TN. 448p.

Miscanthus –6th Yr 14 Mt ha^{-1}

Switchgrass– 6th Yr 8 Mt ha^{-1}

Agricultural Lands- 15- 20 Mt ha^{-1}



Skousen et al. 2017. Growth of Biofuels Crops on Reclaimed Mine Lands of West Virginia. Available at: https://wvmdtaskforce.files.wordpress.com/2018/02/skousen-bioenergy-crops-income-opps-jan-2018.pdf

What to do with Herbaceous Biomass?

2017 U.S. ETHANOL PRODUCTION C… NT

	P…	…ction (mgy)
DuPont Cellul… Nevada, I…		0
Fi… …IA	6	0
…LC, Vero	8	0
…t LIBERTY (POET/DSM), Emmetsburg, IA	20	0
Synata Bio, Inc., Hugoton, KS	25	0

DuPont to sell cellulosic ethanol plant in blow to biofuel-

https://www.reuters.com/article/us-dowdupont-ethanol/dupont-to-sell-cellulosic-ethanol-plant-in-blow-to-biofuel-idUSKBN1D22T5

RFA.2017. Building Partnerships- Growing Markets 2017 Ethanol Industry Outlook. Renewable Fuel Association. Washington D.C. Available at: http://www.ethanolrfa.org/wp-content/uploads/2017/02/Ethanol-Industry-Outlook-2017.pdf

Use of Herbaceous Biomass Crop for BioChar Production

Source	Biomass	Pyrolysis Temp (°C)	Biochar Yield (%)
Mimmo et al. [2] and Michel et al. [3]	Miscanthus	450-500	28-31
Oginni et al. [4]	Switchgrass Miscanthus	500	29-30

Pyrolysis products properties were not significantly influenced by the herbaceous biomass varieties.- Oginni et al. (2017)

[2] T. Mimmo, P. Panzacchi, M. Baratieri, C.A. Davies, G. Tonon, Effect of pyrolysis temperature on miscanthus (Miscanthus × giganteus) biochar physical, chemical and functional properties, Biomass and Bioenergy, 62 (2014) 149-157.
[3] R. Michel, N. Mischler, B. Azambre, G. Finqueneisel, J. Machnikowski, P. Rutkowski, T. Zimny, J.V. Weber, Miscanthus × Giganteus straw and pellets as sustainable fuels and raw material for activated carbon, Environmental Chemistry Letters, 4 (2006) 185-189.
[4] O. Oginni, K. Singh, J.W. Zondlo, Pyrolysis of dedicated bioenergy crops grown on reclaimed mine land in West Virginia, Journal of Analytical and Applied Pyrolysis, 123 (2017) 319-329.

Use of Herbaceous Biomass Crop for Activated Carbon Production

Source	Biomass	Properties	Comment
Ducey et al. [5]	Switchgrass	S_{BET} = 218.7 m^2/g	Steam Activation. Used for soil amendment
Shim et al. [6]	Miscanthus	S_{BET} = 322 m^2/g	Cu Sorption
Kalyani et al. [7]	Turf grass	S_{BET} = 250 m^2/g	Chemical activation ($ZnCl_2$) Used for electrolysis of water for hydrogen production
Our Ongoing Research	Switchgrass	S_{BET} = 1372 m^2/g	Chemical activation (H_3PO_4) Used for electrode materials for energy storage devices

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[5] T.F. Ducey, J.A. Ippolito, K.B. Cantrell, J.M. Novak, R.D. Lentz, Addition of activated switchgrass biochar to an aridic subsoil increases microbial nitrogen cycling gene abundances, Applied Soil Ecology, 65 (2013) 65-72.
[6] T. Shim, J. Yoo, C. Ryu, Y.-K. Park, J. Jung, Effect of steam activation of biochar produced from a giant Miscanthus on copper sorption and toxicity, Bioresource Technology, 197 (2015) 85-90.
[7] P. Kalyani, A. Anitha, A. Darchen, Activated carbon from grass – A green alternative catalyst support for water electrolysis, International Journal of Hydrogen Energy, 38 (2013) 10364-10372.

Potential Economic Benefits

Reclaimed Mine Land Currently Planted with Herbaceous Biomass in WV	About 200 acres
[1]Average dry matter yield after the fourth growing season for • Switchgrass (Kanlow and Bomaster varieties) • Miscanthus **Average**	8,000 kg ha^{-1} $year^{-1}$ 14,000 kg ha^{-1} $year^{-1}$ 11,000 kg ha^{-1} $year^{-1}$
Estimated biochar yield (30% of dry biomass)	3,300 kg ha^{-1} $year^{-1}$
Estimated activated carbon yield ([2]62% of dry biomass)	2,046 kg ha^{-1} $year^{-1}$
Potential market value ($12-13/lb or $29/kg at amazon.com)	$59,334 ha^{-1} $year^{-1}$

There is a potential to produce activated carbon on worth **$4.8 million every year** from herbaceous biomass planted on 200 acres in WV

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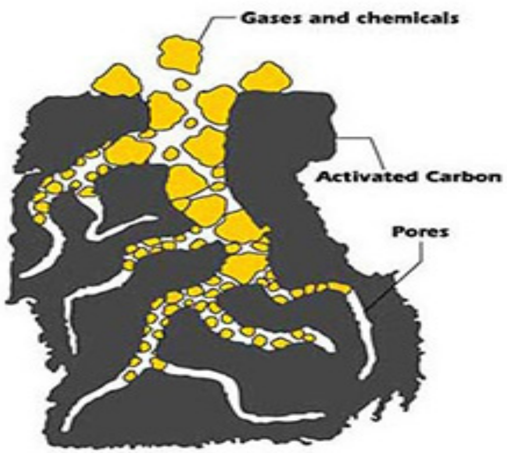
1. Skousen et al. 2017. Growth of Biofuels Crops on Reclaimed Mine Lands of West Virginia. Available at: https://wvmdtaskforce.files.wordpress.com/2018/02/skousen-bioenergy-crops-income-opps-jan-2018.pdf
2. Lu, Q.; Wang, Z.; Dong, C.-q.; Zhang, Z.-f.; Zhang, Y.; Yang, Y.-p.; Zhu, X.-f., Selective fast pyrolysis of biomass impregnated with ZnCl2: Furfural production together with acetic acid and activated carbon as by-products. *Journal of Analytical and Applied Pyrolysis* **2011,** 91, (1), 273-279.

Activated Carbons

"A porous highly adsorptive form of carbon used to remove colour or impurities from liquids and gases, in the separation and extraction of chemical compounds, and in the recovery of solvents." **-** Dictionary.com



Powdered Activated Carbons (PAC)

- particle sizes < 40 µm
- used in slurry reactor
- cannot be reactivated and reused
- faster removal of compounds

Granulated Activated Carbons (GAC)

- particle sizes in the range of 0.5 to 4 mm
- fixed bed adsorbers
- reactivated for reuse

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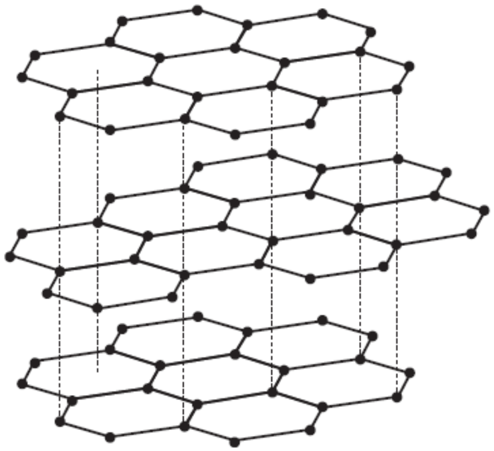
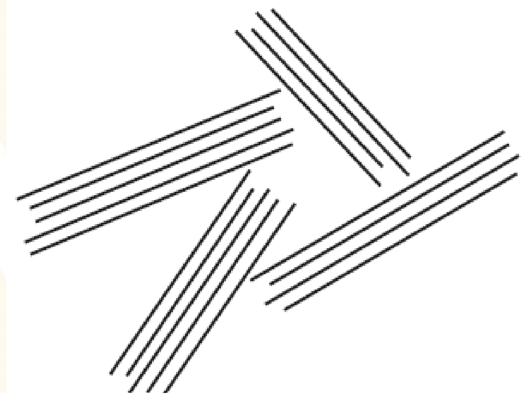
Dictionary.com "activated carbon," in *Dictionary.com Unabridged*. Source location: Random House, Inc.http://www.dictionary.com/browse/activated-carbon. Available: http://www.dictionary.com/. Accessed: February 28, 2018.

Pore Structure of Activated Carbons

- consists of crystallites with a strongly disturbed graphite structure.
- randomly oriented and interconnected by carbon cross-links.
- The micropores are formed by the voids between the crystallites.
- Slit-like pores are found.



Graphite Crystalline Structure



Randomly Oriented Graphite microcrystallites

How Does Activated Carbon Work?

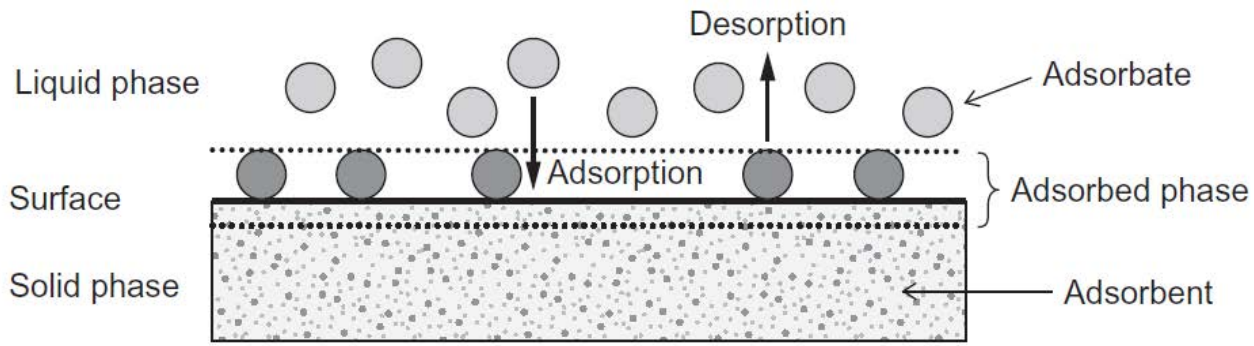


Figure: Basic Single Solute System Adsorption (Worch, 2012)

- Physical Adsorption-
  - It is caused by weak interactions of van der Waals forces (induction forces, dipole-dipole interactions, dispersion forces).

$$H_{ads} < 50\ kJ/mol$$

- Chemical Adsorption-
  - It is caused by chemical reaction between adsorbate and surface sites.

$$H_{ads} > 50\ kJ/mol$$

Sources: Worch, E., *Adsorption technology in water treatment: fundamentals, processes, and modeling*. Walter de Gruyter: 2012

Theoretical Thermodynamic Considerations

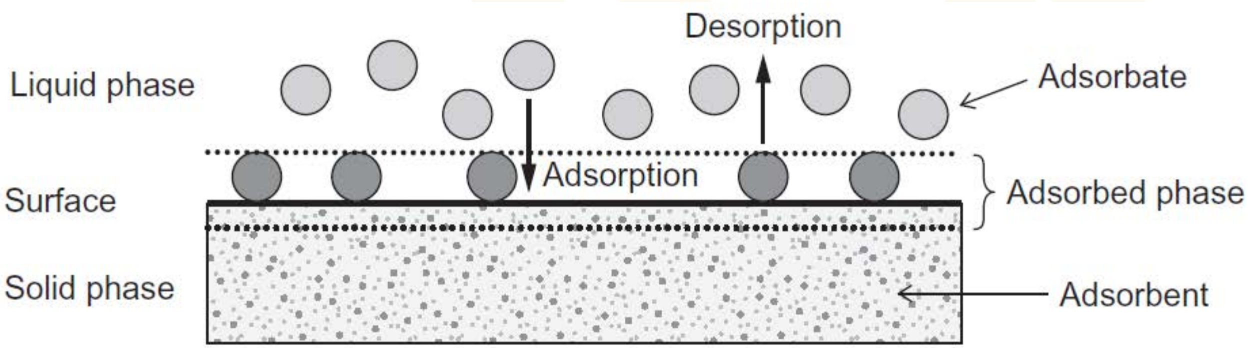


Figure: Basic Single Solute System Adsorption (Worch, 2012)

$$dG = -S\,dT + V\,dp + \sum_i \mu_i\,dn_i + \sigma\,dA$$

$$\sigma = \left(\frac{\partial G}{\partial A}\right)_{T,p,n_i}$$

When adsorption takes place:

$$\sigma_{ws} - \sigma_{as} = \pi > 0$$

G= Gibbs Free Energy
S= Entropy
T= Temperature
p= Pressure
ni= Number of moles
A= Surface
V= Volume
μ= Chemical Potential
σ= Surface free energy
ws= Water-Solid interface
as= Adsorbate solution-solid interface
π= Spreading pressure depends on adsorbate amount

For adsorption to take place, change in free energy must be negative:

$\Delta G_{ads} = \Delta H_{ads} - T\Delta S_{ads} < 0$, since $\Delta S_{ads} < 0$ $\Delta H_{ads} < 0$

Adsorption is a Exothermic process

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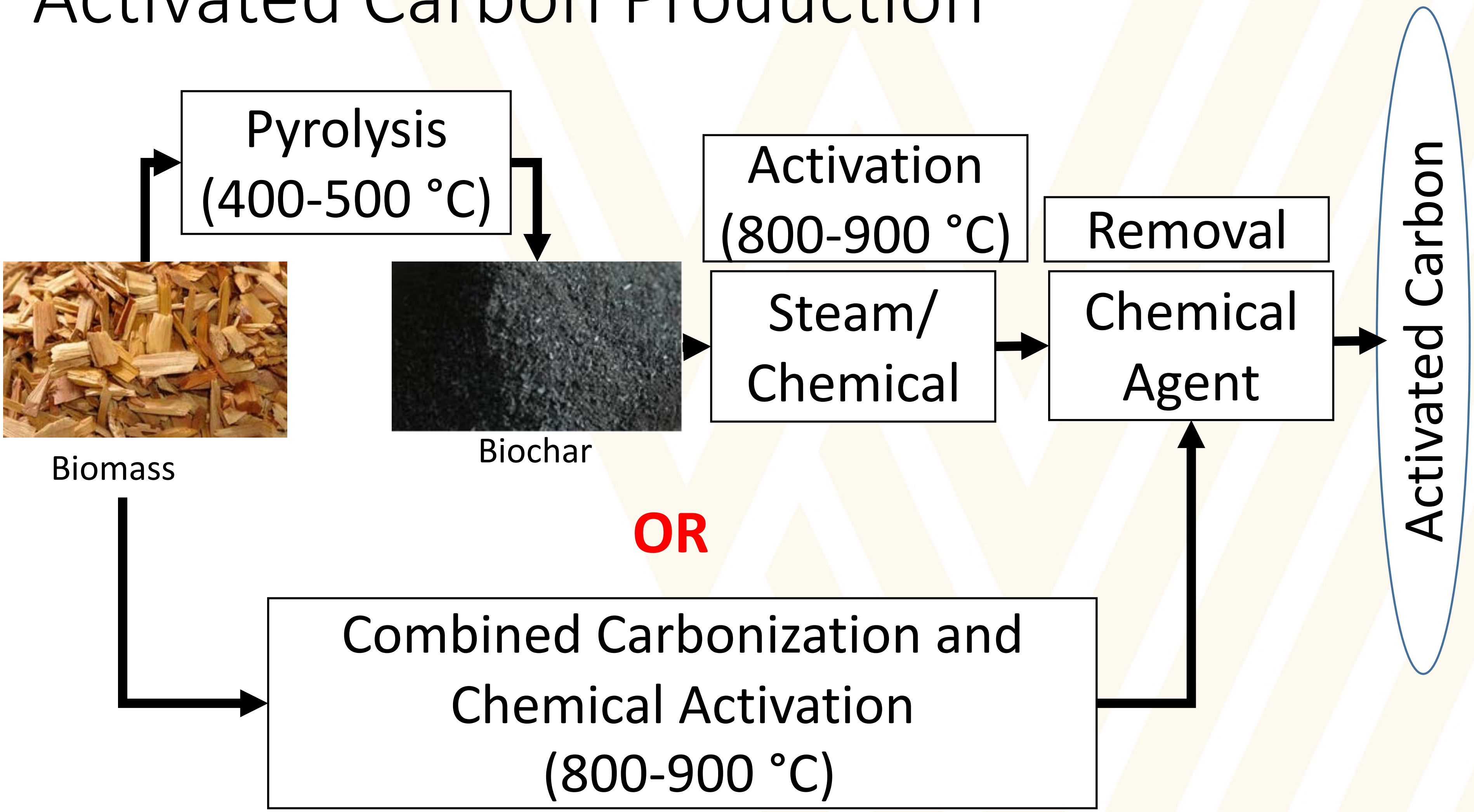
Sources: Worch, E., *Adsorption technology in water treatment: fundamentals, processes, and modeling*. Walter de Gruyter: 2012

Activated Carbon Production



Which Pathway is Better?

Which Pathway is Better?

Source	Specific Gravity	Chemical Agent Impregnated into Biomass/Biochar)	V_{Total} (cm^3/g)	BET Surface Area (m^2/g)
Budinova et al. [1]	0.54 (diffuse porous hardwood)	H_3PO_4 in Birch Biomass	0.618	761
Pereira et al. [2]	-	H_3PO_4 in Cocoa shells Biomass	0.68	1077
Park et al. [4]	0.47 (softwood)	NaOH in Loblolly Pine Biochar	-	1250
Azargohar et al. [5]	0.36 (softwood)	KOH in Spruce Biochar	0.41	837

Literature does not provide clear answer.

[2] R.G. Pereira, C.M. Veloso, N.M. da Silva, L.F. de Sousa, R.C.F. Bonomo, A.O. de Souza, M.O.d.G. Souza, R.d.C.I. Fontan, Preparation of activated carbons from cocoa shells and siriguela seeds using H3PO4 and ZnCL2 as activating agents for BSA and α-lactalbumin adsorption, Fuel Processing Technology, 126 (2014) 476-486.
[4] J. Park, I. Hung, Z. Gan, O.J. Rojas, K.H. Lim, S. Park, Activated carbon from biochar: Influence of its physicochemical properties on the sorption characteristics of phenanthrene, Bioresource Technology, 149 (2013) 383-389.
[5] R. Azargohar, A.K. Dalai, Steam and KOH activation of biochar: Experimental and modeling studies, Microporous and Mesoporous Materials, 110 (2008) 413-421.

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Objective

- To compare the adsorption characteristics of activated carbons (AC) synthesized from herbaceous biomass following two activation methods: 1. Biomass+activation agent-> activated carbon and 2. Biomass-> bio-char+activation agent-> activated carbon.

Switchgrass Harvesting

- **Location**: Alton site, a reclaimed mine land in Upshur County, WV
- **Variety**: Kanlow Switchgrass and Bomaster Switchgrass
- **Time of Harvest**: Fall 2014
- **Productivity at Harvest Site**: 5,200 kg/ha (Dry matter yield)

Alton Soil Characteristics

(Skousen et al., 2017)

- 70% Fines
- pH = 7.5
- EC = 368 µs/cm
- P = 40 mg kg^{-1} soil
- K = 0.2 $cmol_c$ kg^{-1}
- Ca = 3.2 $cmol_c$ kg^{-1}

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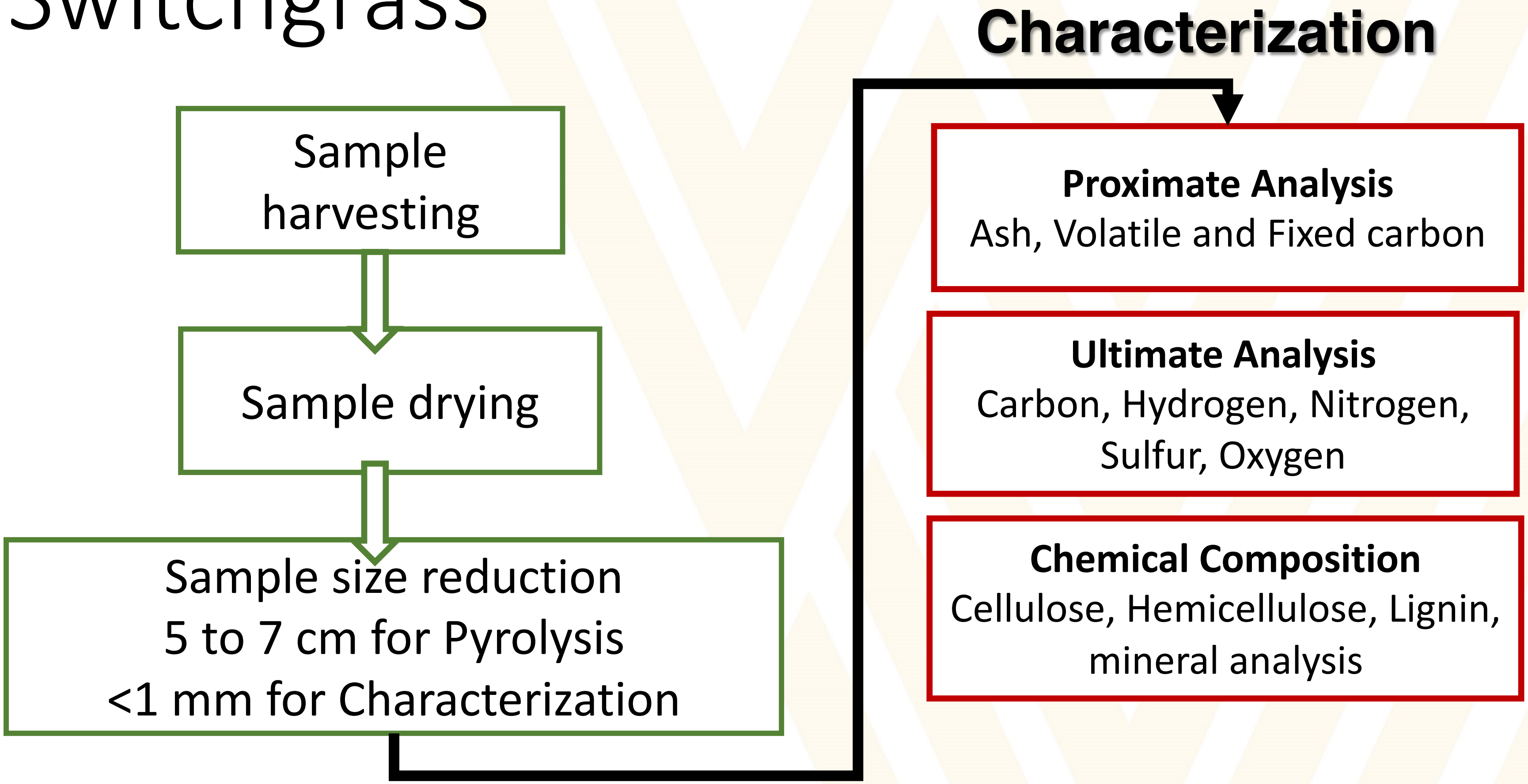
Skousen et al. 2017. Growth of Biofuels Crops on Reclaimed Mine Lands of West Virginia. Available at: https://wvmdtaskforce.files.wordpress.com/2018/02/skousen-bioenergy-crops-income-opps-jan-2018.pdf

Processing and Characterization of Switchgrass

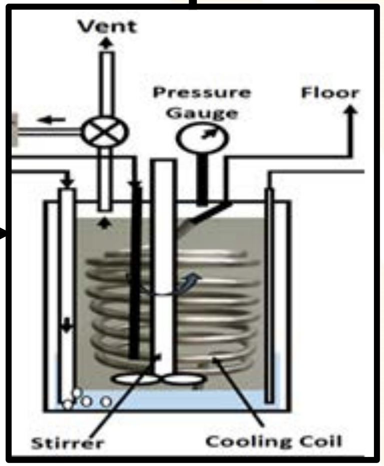


Activated Carbon Production

Biomass

Pyrolysis
500 °C,
30 min,
N_2

Biochar



Impregnation with H_3PO_4
Mixing Ratio: 4:1
Temperature: 85 °C
Duration: 24 h

Drying
103 °C, 24 h

Activation
Temperature: 900 °C
Duration: 30 min
Gas: N_2

Activated Carbon Washing

Drying
103 °C, 24 h

Biomass-Derived and **Biochar-Derived** Activated Carbons (AC)

<u>**Characterizations**</u>: BET Surface Area, Pore Volumes, XPS, Raman Spectra, Scanning Electron Microscopy,
<u>**Testing**</u>- Adsorption Kinetics and Equilibrium

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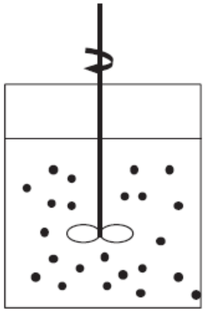
Testing- Adsorption Kinetics and Equilibrium



Slurry Batch Reactor

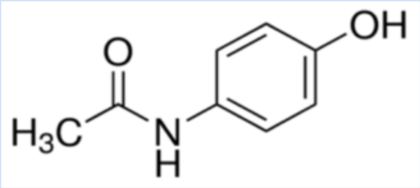
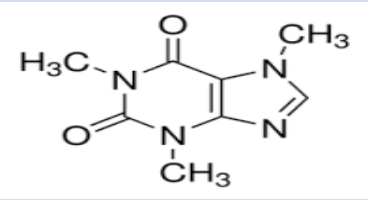
Properties	Acetaminophen	Caffeine
Molecular structure		
Molecular weight (g/mol)	151.16	194.2
Molecular size (nm)	Monomer: **1.19(L)** x 0.75(W) x 0.46(T) Dimer: 1.58 (L) x 1.19(W) x 0.66(T)	**0.98** x 0.87
Use/category	Analgesic, antipyretic, anti-inflammatory drug	Stimulant

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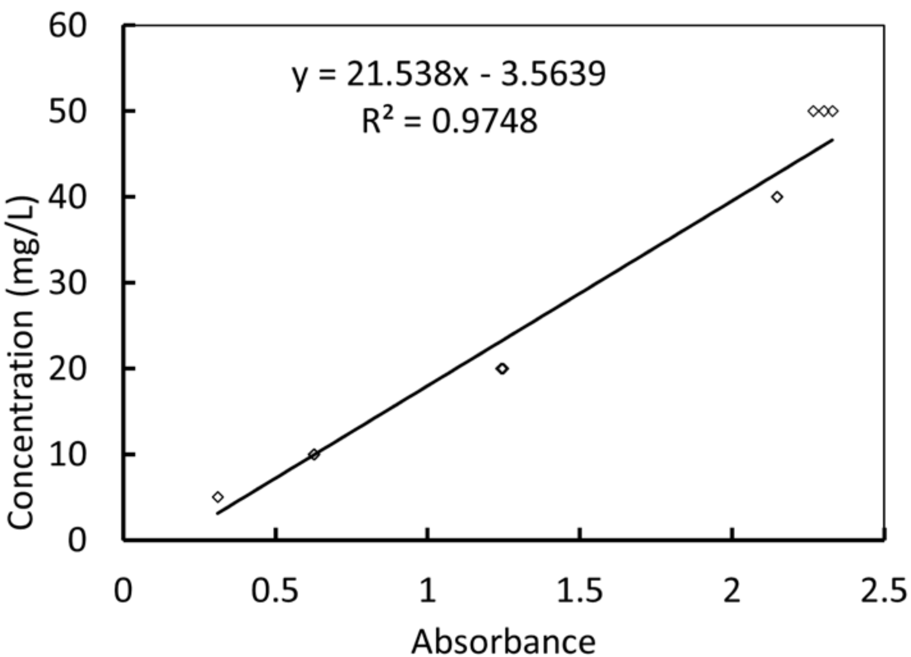
Image Sources:
http://www.thrombocyte.com/is-acetaminophen-a-blood-thinner/
https://www.cbsnews.com/media/things-you-should-know-about-caffeine/

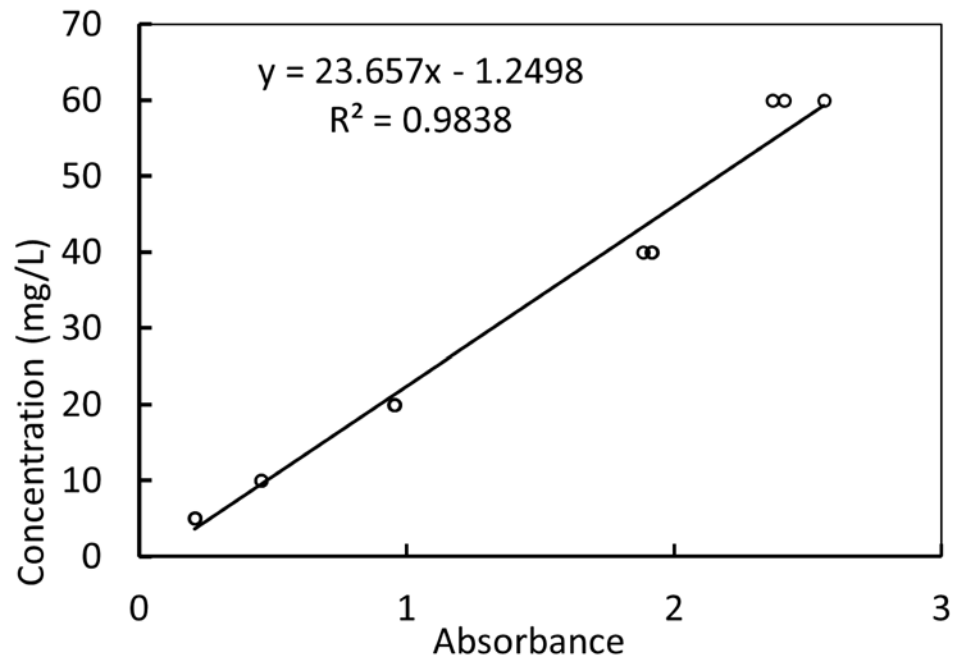
Concentration Measurement using UV–VIS Spectrophotometer

- Standard solutions (5 to 50 ppm) were scanned between 200 nm and 400 nm.
- Deionized water as a blank.
- Peak absorbance at 242 nm and 274 nm were recorded for Acetaminophen and Caffeine, respectively.
- Calibration was prepared pe

Accuracy of Measurements= ±1.2%



Calibration for **Acetaminophen**



Calibration for **Caffeine**

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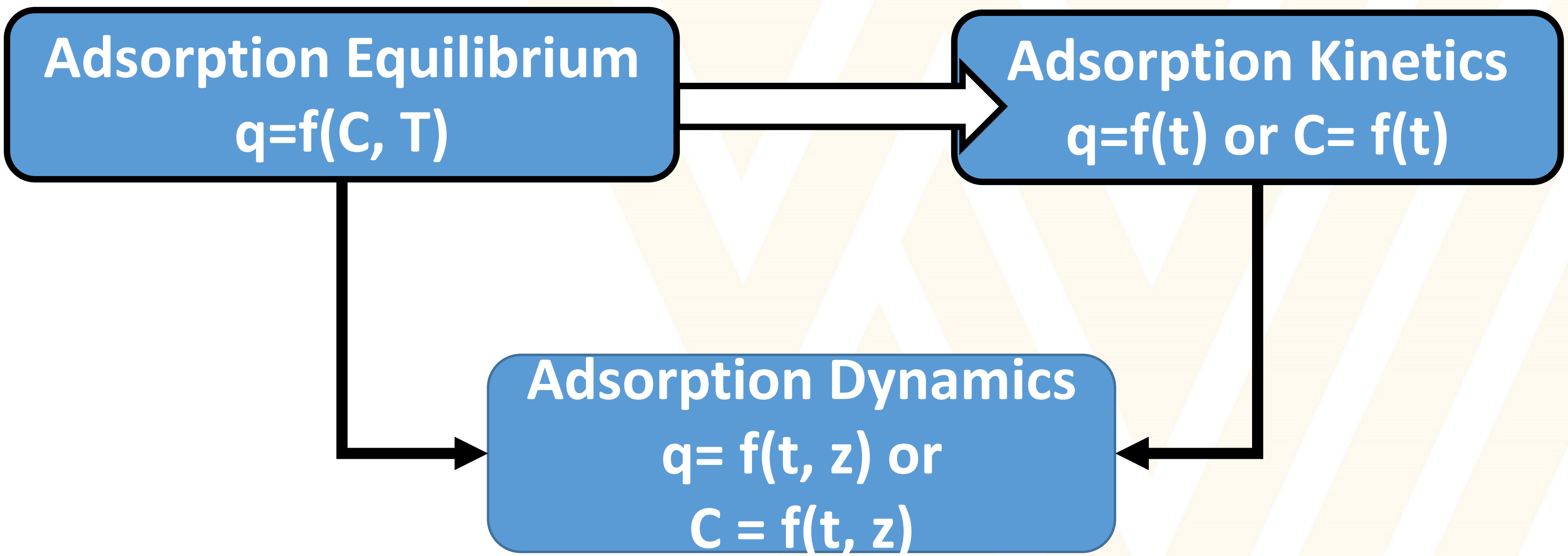
Adsorption Mass Transport Theory

$$\textbf{Adsorbent Loading (q)} = \frac{Adsorbed\ Amount}{Asorbent\ Mass}$$



Adsorbate Concertation= C; Temperature= T; Space= z; Time= t

Sources: Worch, E., *Adsorption technology in water treatment: fundamentals, processes, and modeling*. Walter de Gruyter: 2012

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Adsorption Testing- Single Solute System

Adsorption Kinetics

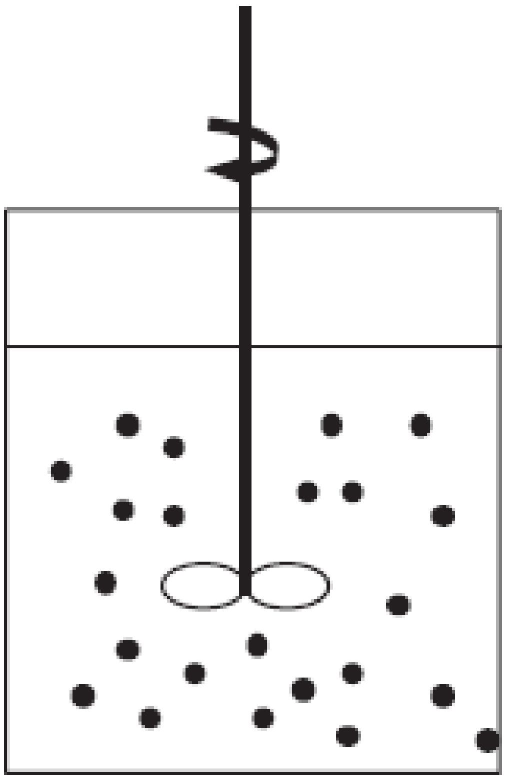
q= f(t)

- Solute Concertation= 40 ppm
- Solution pH= 6.21 (Caffeine) and 5.97 (Acetaminophen)
- Durations= 0.25, 0.50, 1, 3, 5, 7, and 9 hours
- Temperature= 25 °C
- Adsorbent Loading= 10 mg
- Solution volume= 40 ml

Adsorption Equilibrium

q=f(C, T); T= Constant

- Solute Concentrations (ppm)=10, 20, 30, 40
- Solution pH= 6.21 (Caffeine) and 5.97 (Acetaminophen)
- Duration= 5 hours
- Temperature= 25 °C
- Adsorbent Loading= 10 mg
- Solution volume= 40 ml



Slurry Batch Reactor

Sources: Worch, E., *Adsorption technology in water treatment: fundamentals, processes, and modeling*. Walter de Gruyter: 2012

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Adsorption Kinetics- The pseudo-second order kinetic model

$$q_t = \frac{C_o - C_t}{W} V$$

C_o = initial concentration (ppm),
C_t = concentration at time t (ppm),
V = volume of the adsorbate solution (ml),
W = weight of the activated carbon used (mg), and
q_t = amounts of the adsorbate adsorbed at time t (mg/g)

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

q_e= amounts of the adsorbate adsorbed at equilibrium (mg/g),
t= *time (min)*
k_2= equilibrium rate constant (g/mg.min),
k_2 and q_e can be estimated from the intercept and slope, respectively, of the plot of t/q_t versus t,
h= $k_2 . q_e^2$ = the initial adsorption rate (mg/g.min),
$t_{1/2}$ = the time required for the adsorbent to uptake half of the adsorbate amount= $t_{1/2} = \frac{1}{k_2 q_e}$

A.S. Mestre, J. Pires, J.M.F. Nogueira, A.P. Carvalho, Activated carbons for the adsorption of ibuprofen, Carbon, 45 (2007) 1979-1988.

Adsorption Equilibrium

Langmuir isotherm model

$$q_e = \frac{q_m \cdot b \cdot C_e}{1 + b \cdot C_e}$$

q_e = equilibrium quantity adsorbed (mg/g)
C_e =equilibrium concentration of adsorbate (ppm)
q_m = maximum adsorption capacity (mg/g)
b = Langmuir constant (1/ppm)
q_m and b can be determined from the linear plot of C_e/q_e versus C_e.

Freundlich Isotherm Model

$$ln q_e = ln K_f + \frac{1}{n} ln C_e$$

K_f = strength of adsorption
n = energetic heterogeneity of adsorbent surface
The values of n and K_f can be obtained from the slope and intercept of the linear plot of lnq_e versus lnCe.

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Results- Biomass Characterization

Characteristics	Kanlow Switchgrass (This Study)	Kanlow Switchgrass in Iowa (Lemus et al. 2002)
Chemical Composition		
• Cellulose (%,dry matter)	36.7	38.5
• Hemicellulose (%,dry matter)	31.6	32.8
• Lignin (%,dry matter)	**9.3**	**6.0**
• Others (%,dry matter)	19.4	17.3
Elemental Composition		
• Carbon	47.03	48.0
• Hydrogen	6.45	5.4
• Nitrogen	0.53	-
• Sulfur	0.54	-

Lemus, R.; Brummer, E. C.; Moore, K. J.; Molstad, N. E.; Burras, C. L.; Barker, M. F., Biomass yield and quality of 20 switchgrass populations in southern Iowa, USA. *Biomass and Bioenergy* **2002,** *23* (6), 433-442.

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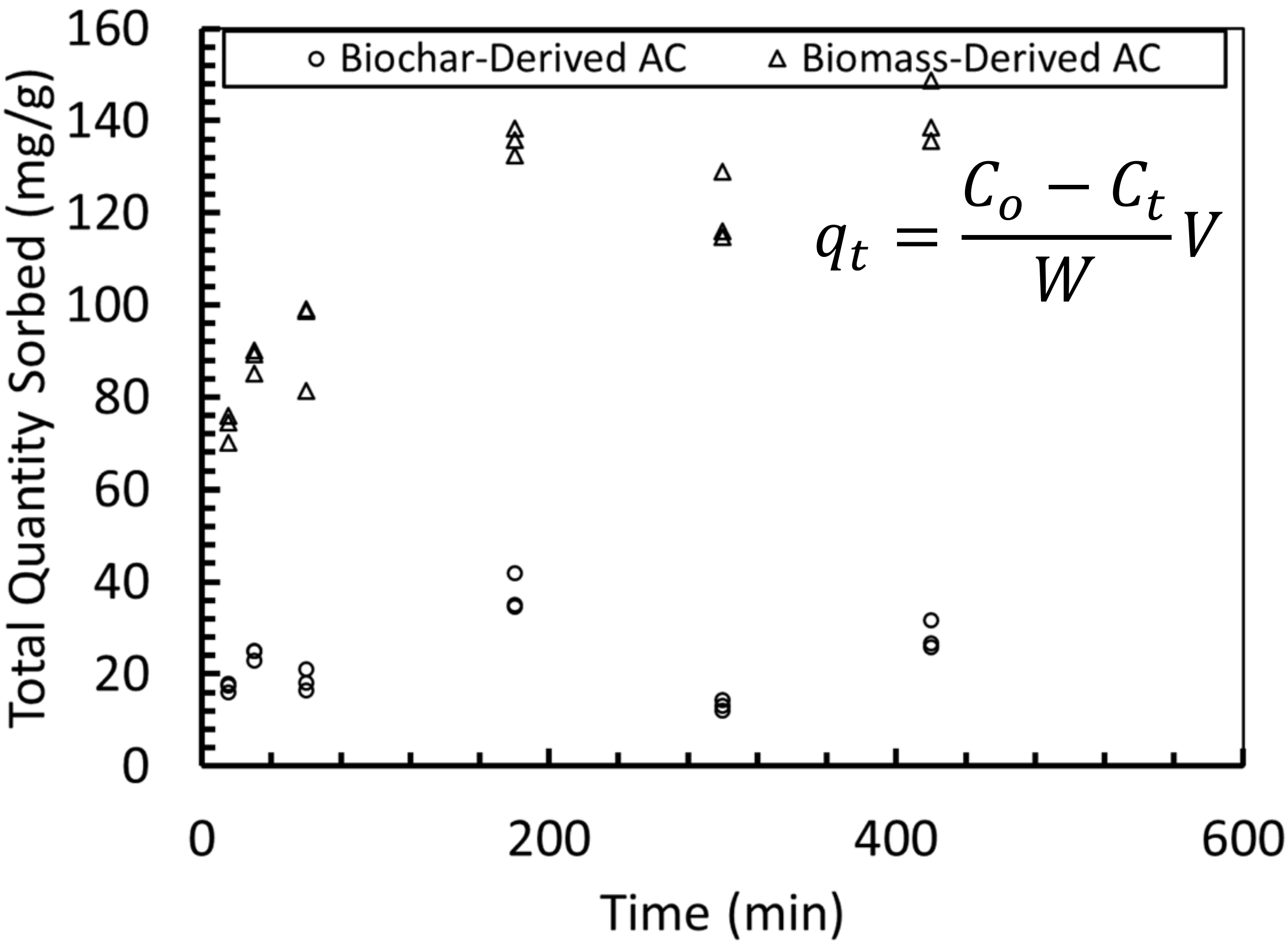
Results- Biomass Characterization

Characteristics	Kanlow Switchgrass (This Study)	Kanlow Switchgrass in Iowa (Lemus et al. 2002)
Higher Heating Value (MJ/kg)	19.3	16.4
Proximate Composition		
• Volatile (%,dry matter)	80.6	81.5
• Fixed Carbon (%,dry matter)	17.0	13.9
• Ash (%,dry matter)	3.06	5.4
Ash Composition (oxides)		
• Phosphorus (%,dry matter)	0.02	0.05
• Potassium (%,dry matter)	**0.36**	**0.09**
• Calcium (%,dry matter)	0.26	0.11
• Magnesium (%,dry matter)	0.09	0.05

Lemus, R.; Brummer, E. C.; Moore, K. J.; Molstad, N. E.; Burras, C. L.; Barker, M. F., Biomass yield and quality of 20 switchgrass populations in southern Iowa, USA. *Biomass and Bioenergy* **2002,** *23* (6), 433-442.

Results- Adsorption Kinetics



$$q_t = \frac{C_o - C_t}{W} V$$

Sorption of **Caffeine** over time on the two Activated Carbons

Results- Adsorption Kinetics

The pseudo-second order kinetic model

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e} t$$

Model parameters	Caffeine	
	Biochar derived AC	Biomass derived AC
q_e (mg/g)	**21.74**	**140.85**
k_2 (g/mg.min)	7.08×10^{-3}	0.30×10^{-3}
h (mg/g.min)	3.35	6.58
	Acetaminophen	
q_e (mg/g)	**43.10**	**86.21**
k_2 (g/mg.min)	0.49×10^{-3}	1.29×10^{-3}
h (mg/g.min)	0.92	9.59

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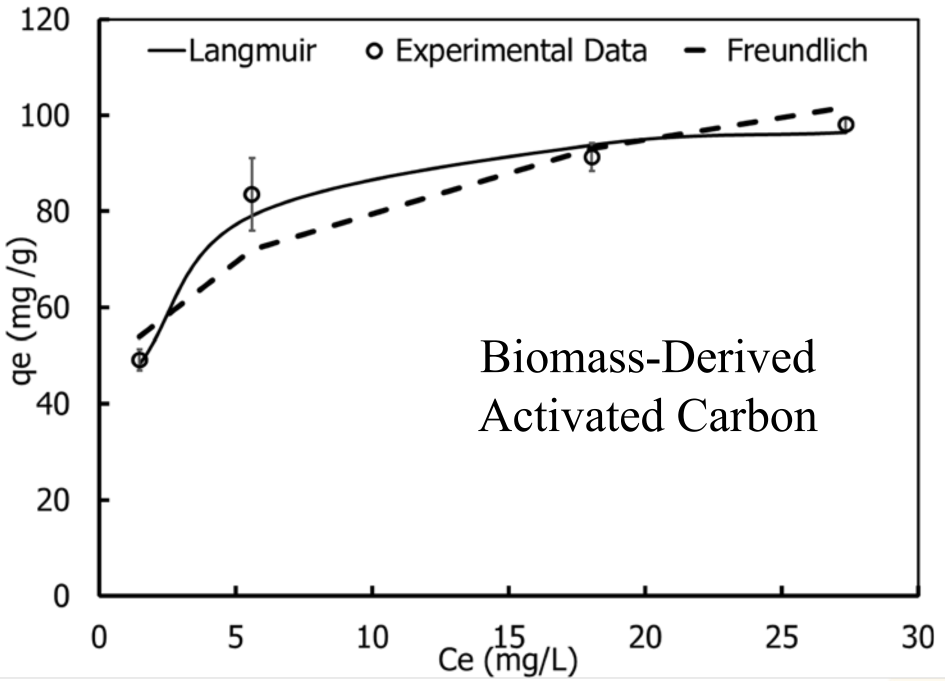
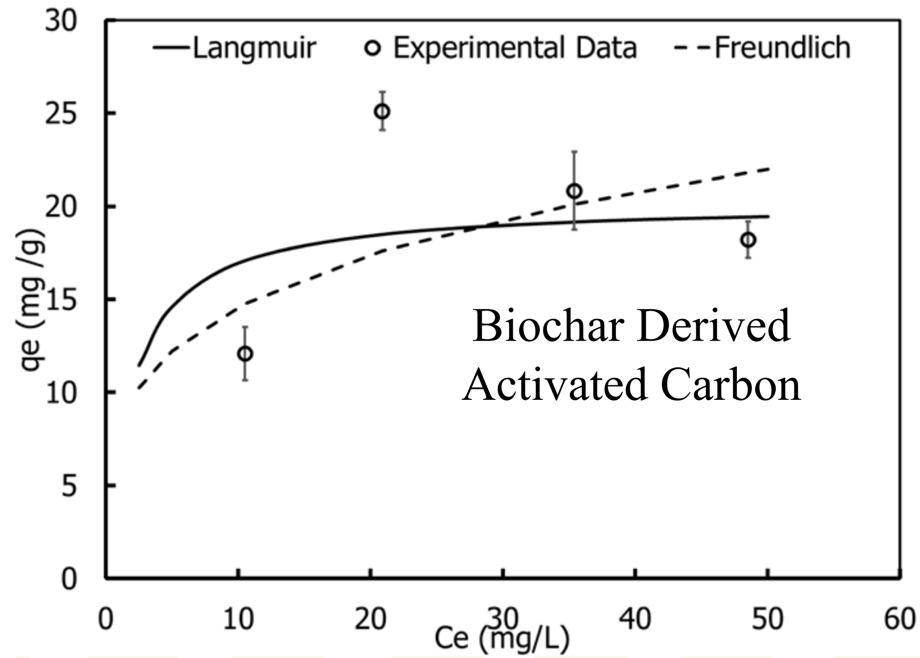
Results- Adsorption Equilibrium-Caffeine





- Langmuir isotherm model provides the better fit for the adsorption of caffeine than the Freundlich model.

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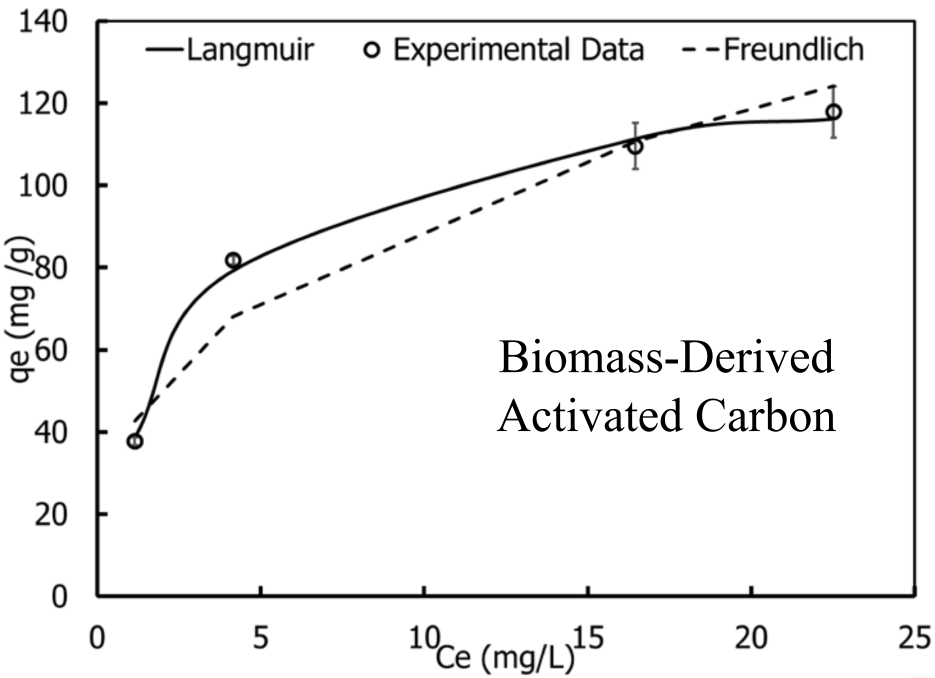
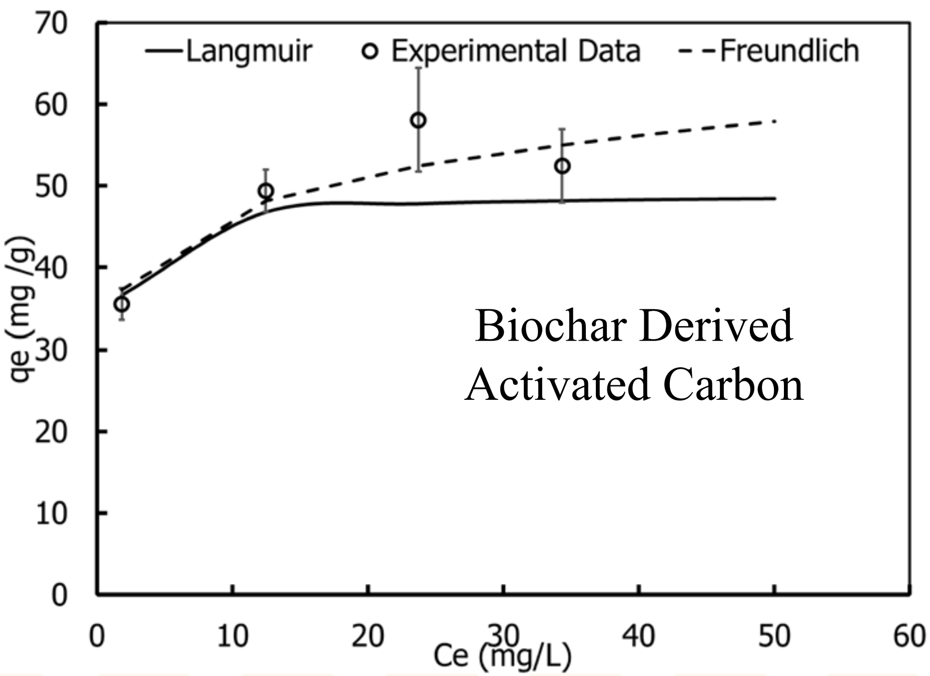
Results- Adsorption Equilibrium-Acetaminophen



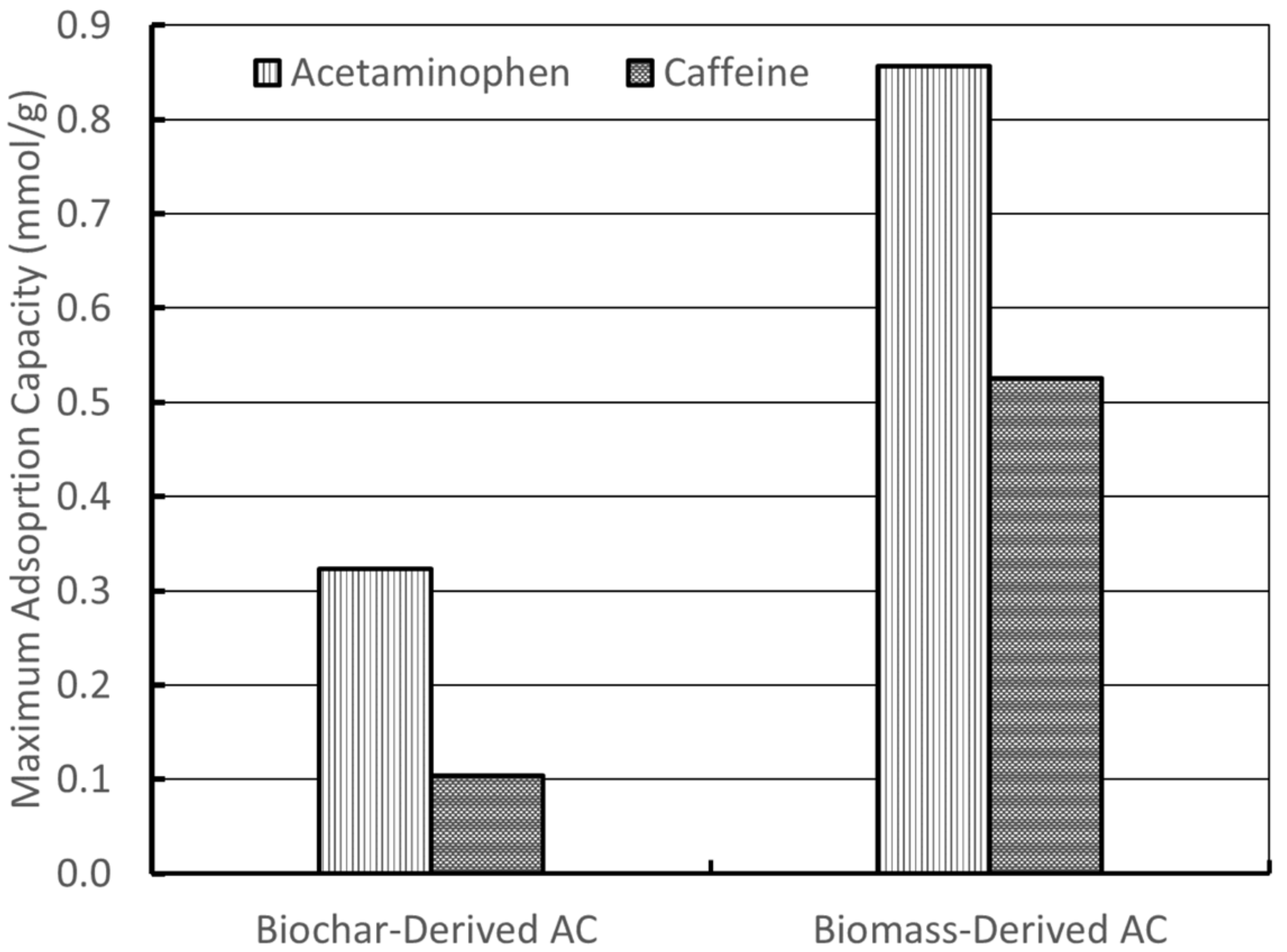


- Langmuir isotherm model provides the better fit for the adsorption of Acetaminophen than the Freundlich model.

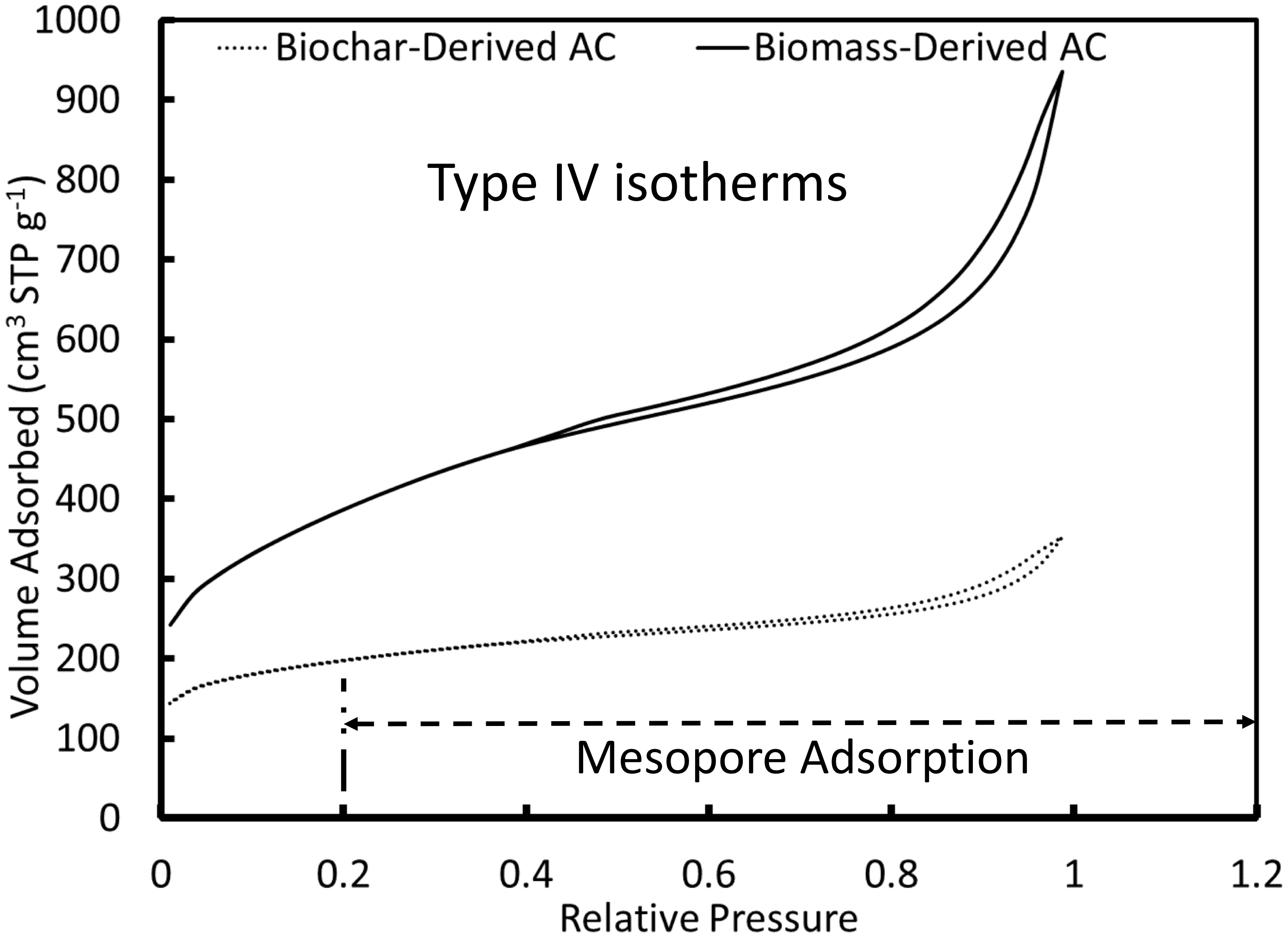
Results- Adsorption Equilibrium- Model

Activated Carbon	A
Biochar-Derived AC	C
	A
Biomass-Derived AC	C
	A

q_m= Maximum m
b= Langmuir cons



Results- Pore Characteristics



Nitrogen adsorption isotherms at 77k

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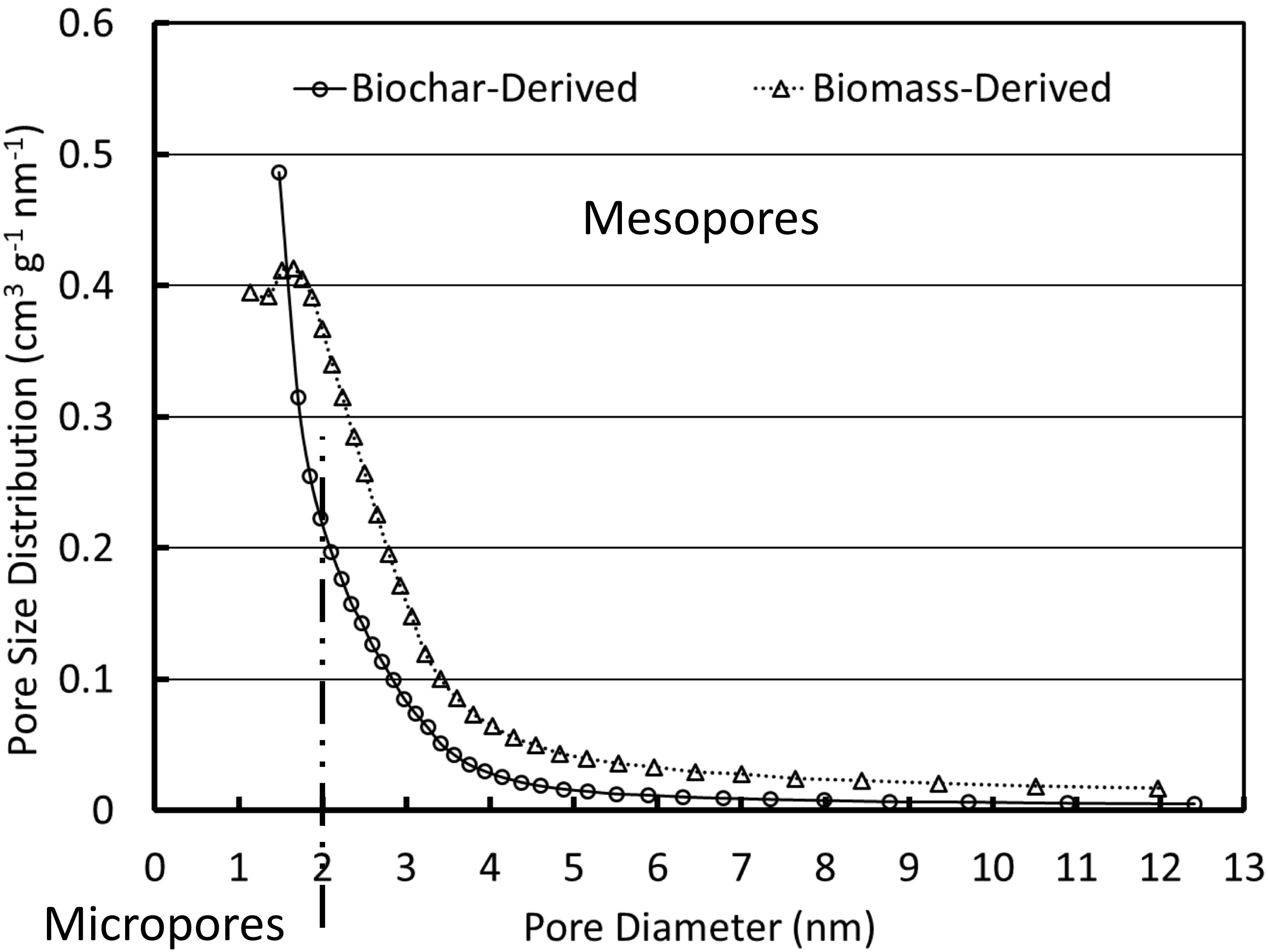
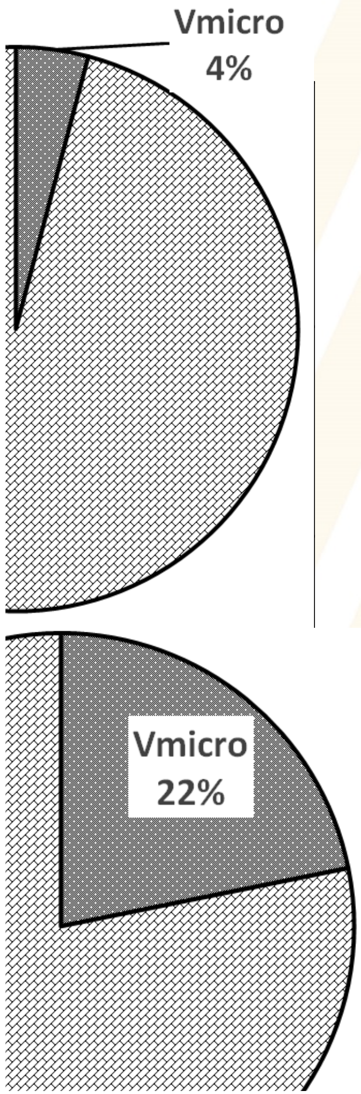
Results- Pore Characteristics





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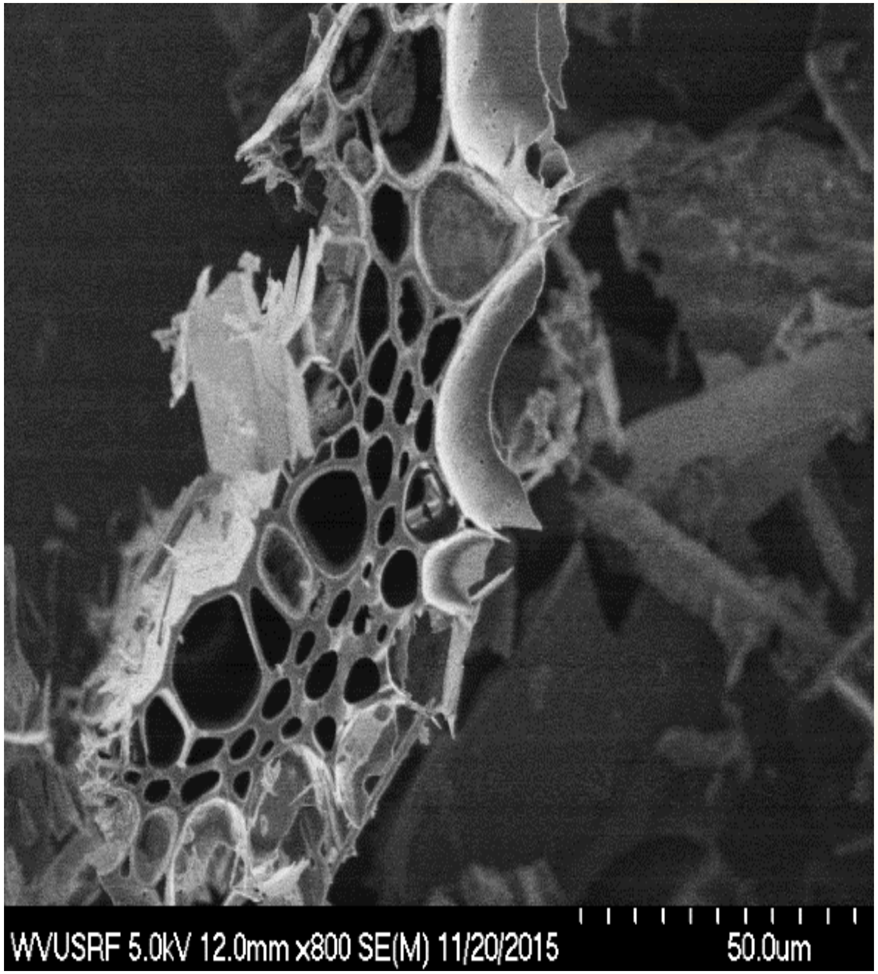
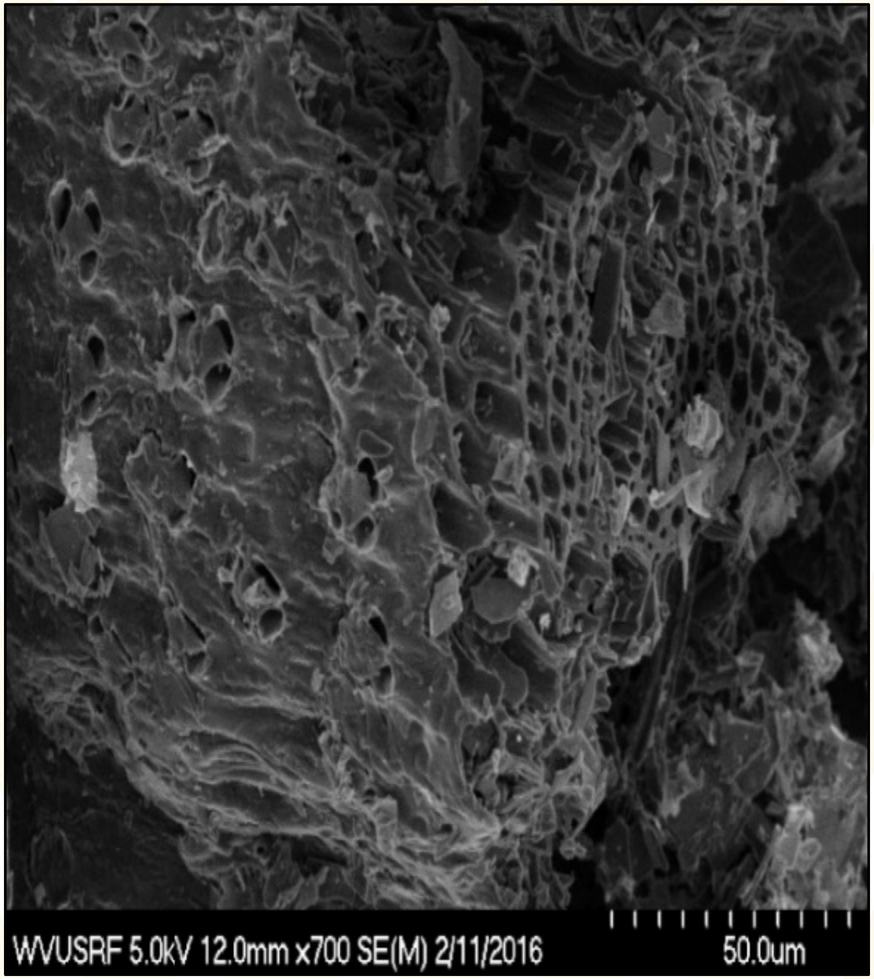
For effective adsorption, the pore diameter of the activated carbon has to be 1.7 to 2 times bigger than the adsorbate dimension.

Results- Ultrastructure of ACs



Biomass-Derived Activated Carbon



Biochar-Derived Activated Carbon

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Results- Surface Chemistry: XPS

Atomic concentration of functional groups on activated carbon surface

Activated Carbon	Atomic Concentration (%)					
	C 1s	O 1s	Si 2p	Al 2p	P 2p	Fe 2p
Biomass-Derived AC	79.68	15.03	0.61	0.57	3.36	0.75
Biochar-Derived AC	74.14	18.75	1.26	1.83	4.03	-

- Oxygen content makes the activated carbon more hydrophilic in nature.

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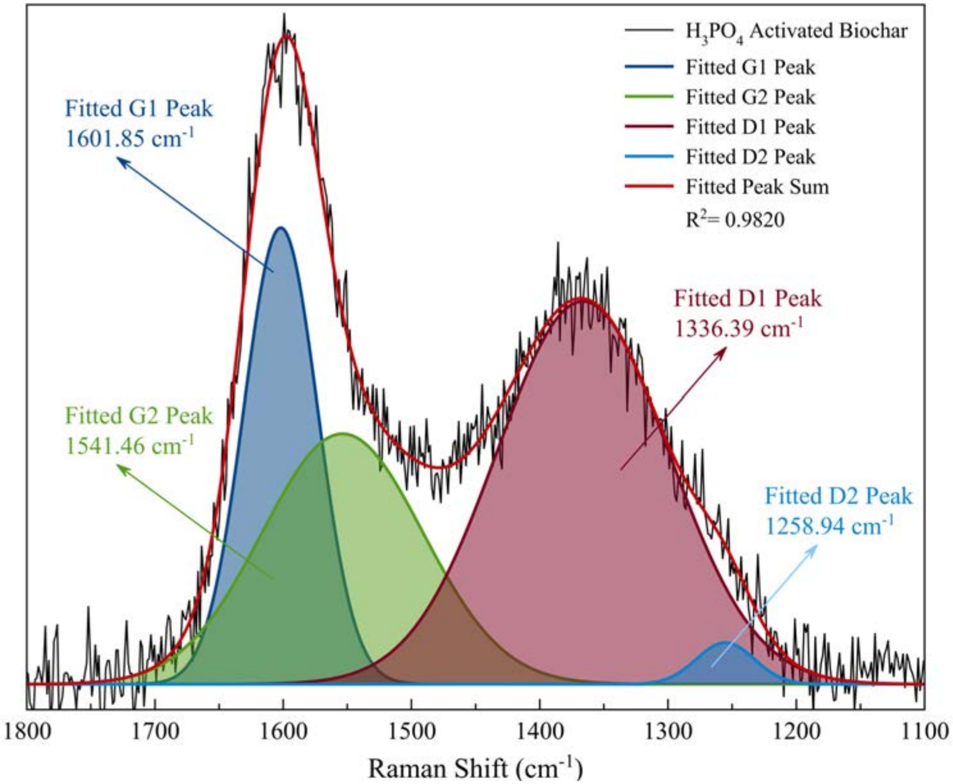
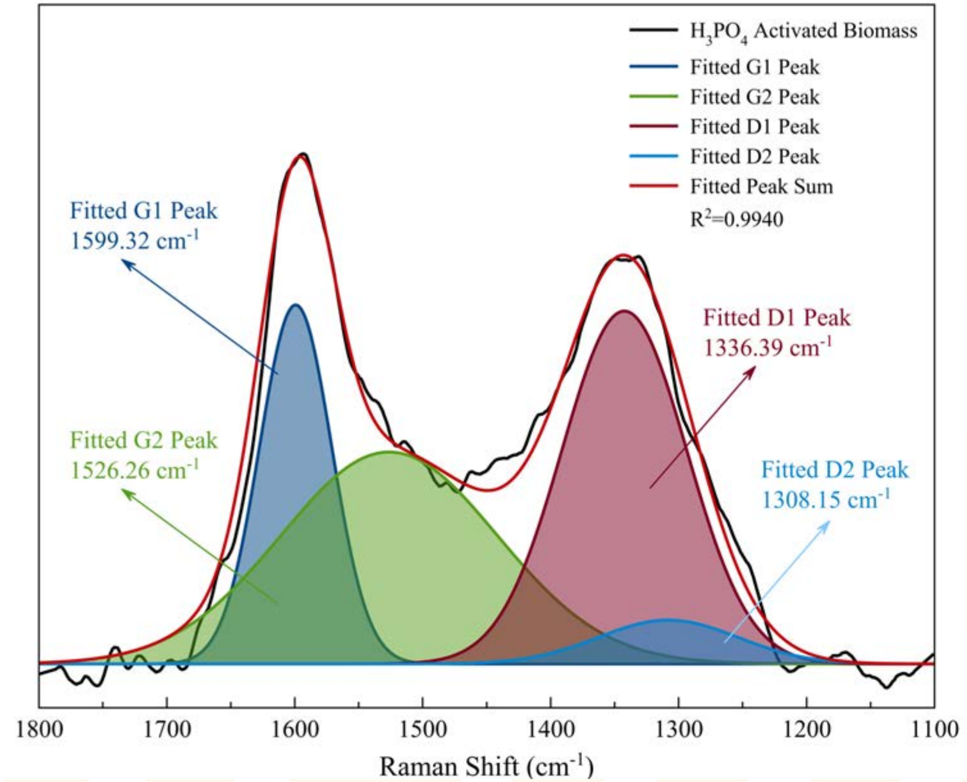
Puziy, A. M., et al. "XPS and NMR studies of phosphoric acid activated carbons." *Carbon* 46.15 (2008): 2113-2123.

Results- Surface Chemistry: Raman Spectra

Biochar-Derived AC



Biomass-Derived AC



- Heterogeneous carbon microstructure
- The basal plane were similar.
- The amorphous sp^2 carbon was more in Biomass-Derived AC.

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Shimodaira, N., and A. Masui. "Raman spectroscopic investigations of activated carbon materials." *Journal of Applied Physics* 92.2 (2002): 902-909.

Conclusions

1. Biomass-derived activated carbon showed 5-folds more adsorption for caffeine and 2.5-folds more adsorption for acetaminophen than biochar-derived activated carbons.

2. The high adsorption was dues presence of more mesopore volume and associated surface area on Biomass-Derived AC.

3. The biomass-derived activated carbon particles showed more disordered SP^2 carbon cluster, which is associated with presence of more amorphous region.

Take Home Message- The impregnation of activation agent (H_3PO_4) directly into biomass resulted in activated carbons with better adsorption capability for both caffeine and acetaminophen than that produced from biochar impregnation.

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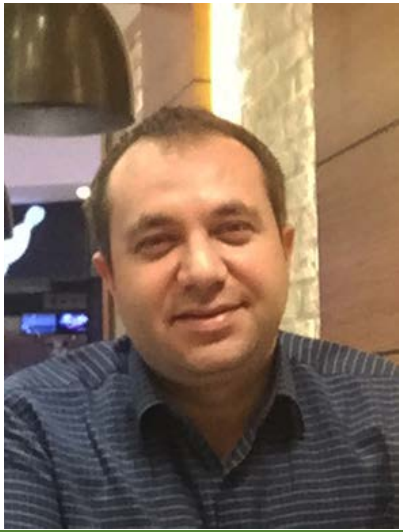

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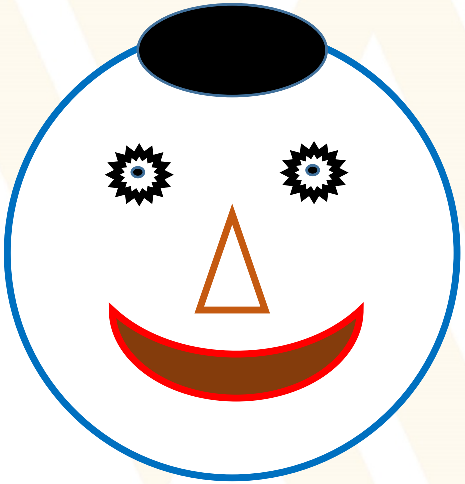
Thank you for your undivided attention!!!!!



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Key Adsorbent Characteristics

Densities: Material Density, Particle Density and Bed Density

Porosities: Particle Porosity and Bulk Porosity

External Surface Area: Little importance

Internal Surface Area: How much of it being used?

Pore Size Distributions: High micropore volume = high adsorbent capacity?

Surface Chemistry: Surface oxides (acidic and basic) and Point of zero charge

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Sources: Worch, E., *Adsorption technology in water treatment: fundamentals, processes, and modeling*. Walter de Gruyter: 2012