Apr 20th, 1:00 PM - 3:00 PM

Modeling of surface area and pore volume of activated carbons prepared from renewable and low cost precursors

Kacey A. Carido
Sacred Heart University

Amanda C. Balesano
Sacred Heart University

Christopher J. Domville
Sacred Heart University

Aaron M. Schwartz
Sacred Heart University

Krisjana Seferi
Sacred Heart University

See next page for additional authors

Follow this and additional works at: https://digitalcommons.sacredheart.edu/acadfest

First and Last Name/s of Presenters

Kacey A. Carido, Amanda C. Balesano, Christopher J. Domville, Aaron M. Schwartz, Krisjana Seferi, and Penny A. Snetsinger

Modeling of surface area and pore volume of activated carbons prepared from renewable and low cost precursors

Amanda Balesano, Christopher Domville, Aaron Schwartz, Kristjana Seferi, Kacey Carido
Dr. Eid Alkahtib, Dr. Penny Snetsinger
Sacred Heart University Department of Chemistry, Fairfield, CT

Abstract

In this study, methylene blue and iodine numbers were used to optimize the conditions of preparation of activated carbons from renewable and low cost precursors. Standard methods of determining iodine number and methylene blue adsorption were applied. These were used to model surface area and pore volume. Each carbon prepared under different experimental conditions was characterized with the goal of using the data as a response factor in the determination of optimum preparation conditions for each carbon.

Introduction

Activated carbon is and has been one of the most efficient agents applied in water and wastewater treatment. Commercially produced activated carbon however, can be expensive and is normally produced from non-renewable sources. Our research group is interested in generating activated carbon from renewable and low cost materials that occur naturally and are normally discarded in many regions of the world including, olive pits, date pits, acorn shells, corn husks and coconut shells. Factors influencing the efficiency of the activation process include activation temperature (600 ^oC, 700 ^oC, and 800 ^oC), activation time (1 hr, 2 hr, and 3 hr), and nitrogen flow rate (150 ml/min, 250 ml/min, and 350 ml/min). A factorial design of experiment with ANOVA (Analysis of Variance) analysis is an efficient method of evaluating the effect of these factors and possible synergistic relationships between factors.   As part of the ANOVA analysis, a response variable of interest, in this case, "adsorption capacity" must be identified. Various methods of determining adsorption capacity have been used. The two most common are **iodine number (IN)** and the **Methylene blue number (MBN)**, the amount of iodine or Methylene blue (a heterocyclic aromatic dye) adsorbed on activated carbon under defined conditions. Iodine number typically identified the small pore (<2 nm) capacity of activated carbon and therefore reflects its ability to adsorb small substances and has a standardized procedure (ASTM-D4607) Because effluents from textile industries may contain high concentrations of commercial dyes, methylene blue number which is related to the macro and meso pore capacity of activated carbon may be a better measure of adsorption capacity for wastewater treatment in some circumstances. Unfortunately, there is no standardized testing procedure for methylene blue number. The goal of this project is to develop a method for determining MBN and comparing it to IN and a combination of both factors in order to optimize preparations of activated carbons.

Methods & Materials

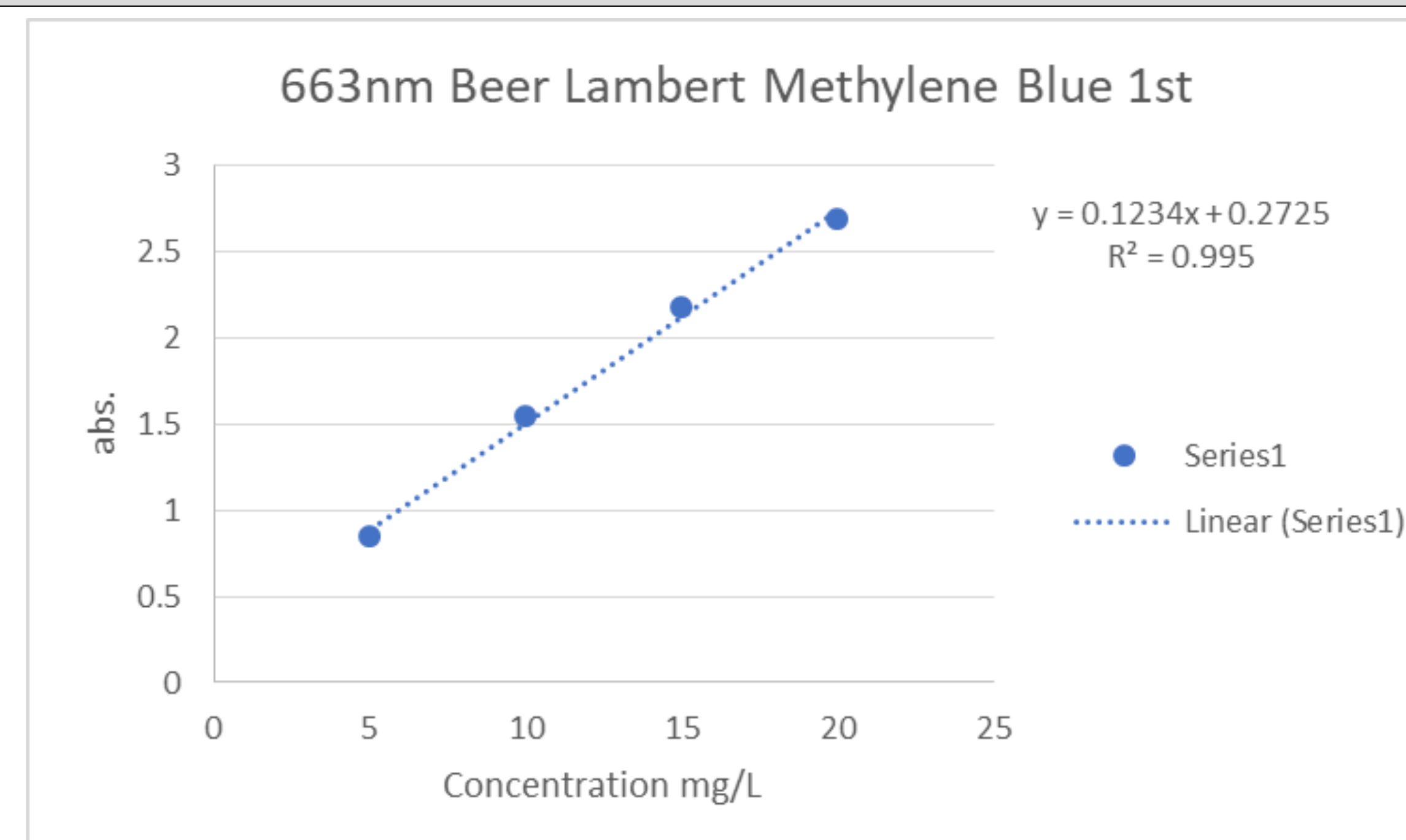Activated Carbons: (supplied by CalgonCarbon, Pittsburgh, PA, USA)

Filtrasorb 600M   SGL 8x30   AP 4-60

Student Prepared Activated carbon-Date Pits
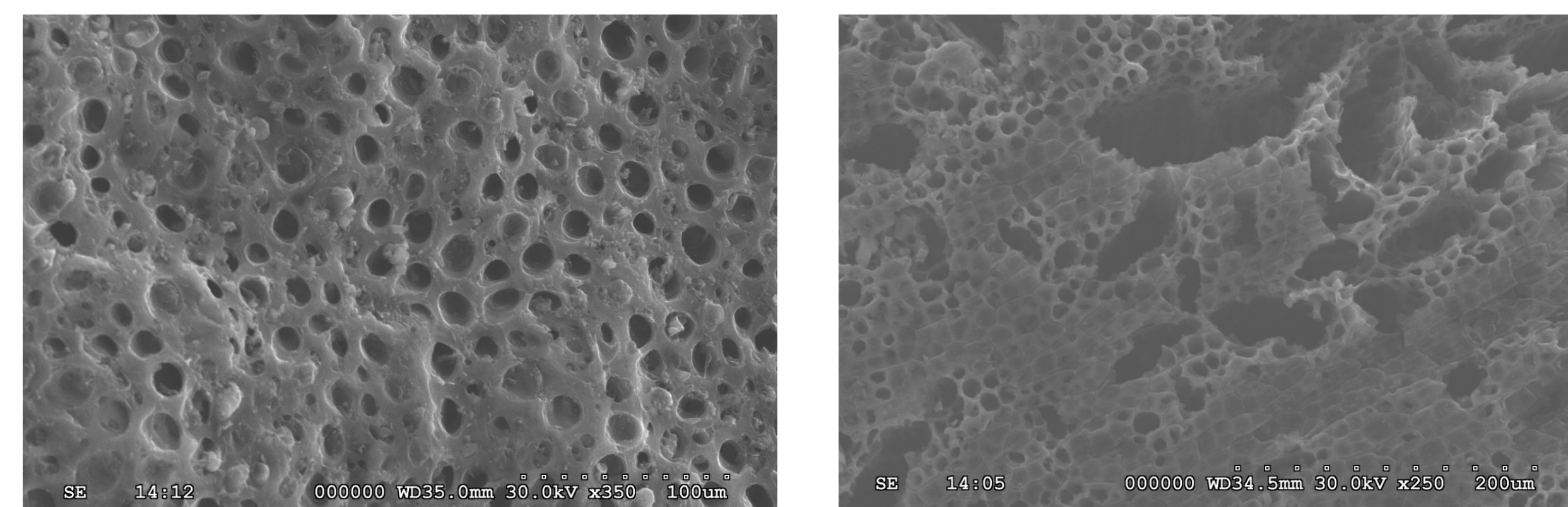
Date Pits Activated Carbon (25% $ZnCl_2$, 800 ^oC and 200 ml/min Nitrogen flow

Date Pits Activated Carbon (25% $ZnCl_2$, 800 ^oC and 200 ml/min Nitrogen flow rate)

Experimental

663nm Beer Lambert Methylene Blue 1st

y = 0.1234x + 0.2725
R^2 = 0.995

Create stock solution of methylene blue
1. Weigh out 200mg (.2g) of methylene blue ($C_{16} H_{18} 〚ClN〛_3 S \cdot xH_2 O$)
2. Place solid methylene blue into 1000 mL volumetric flask
3. Dilute with distilled water up to the line
4. Pour into 1000 mL amber bottle and label
5. Obtain (9) 100 mL amber bottles
6. Place stir bar into each bottle
7. Label each bottle with the following: 30A, 30B, 60A, 60B, 70A, 70B, 80A, 80B, and blank (methylene blue)

Carbon sample preparation
1. Grind carbon with pestle and mortar until it appears as a powdery substance
2. Place grinded carbon onto 2 layer sifter
3. Collect sifted carbon into a beaker (note: you should only grind and sift carbon enough for one sample run)
4. Weighing out sifted carbon
5. Weigh out 2 of each of the following weights and place in appropriate bottle
6. 30 mg (.0300 g) 30A & 30B, 60 mg (.0600 g) 60A & 60B, 70 mg (.0700 g) 70A & 70B, and 80 mg (.0800 g) 80A & 80B
7. Place 100 mL of methylene blue in each bottle
8. Use pipette to obtain methylene blue from 100 mL graduated cylinder and splash over areas that have excess carbon on weighing boat (repeat for each sample) Note: blank bottle should just have 100 mL of methylene blue and stir bar
9. Cap each bottle place each on stir tray, turn stirring level between 2 and 3, and allow stirring for 24 hours

Collecting Data (after 24 hours)
1. Turn off stir tray and obtain (10) 15 mL centrifuge tubes
2. Use pipettes to fill each tube with 10 mL of each sample (include blank and 1 tube of distilled water)
3. Place samples on test tube holder
4. Take samples to the biochemistry lab and centrifuge
5. Distribute each centrifuge tubes evenly in order for the centrifuge to be balanced
6. Close lid (should hear a clicking sound for security), turn switch to "lock"
7. Turn timer to 10 mins and turn on centrifuge
8. After 10 mins turn off timer on centrifuge, wait until the speed hits "0000", switch to "unlock", press button to open switch, and turn off centrifuge
9. Take samples to instrumental lab
10. Turn on computer connected to UV Spectrometer and turn on switch of UV

Spectrometer
1. Open up program (UV Prob 2.35) on computer
2. Connect instrument and set absorbance to 663 nm
3. Fill (1) cuvette with distilled water and click "auto zero"
4. Fill (9) cuvettes with each sample from centrifuge tubes
5. Take absorbances, and record data
6. Use data for calculations to create Beer Lambert Plot

Results & Discussion

After a few trials we were able to determine the three methods: methylene blue, iodine number, and surface area are best for optimizing commercial carbons. Using these parameters, each carbon resulted in a varying degree of successes as seen in the table below.

Type of Carbon	Methylene Blue Number	Iodine Number	Surface Area from methylene blue iodine numbers (
OLC 12x30	n/a		
F100	n/a	816	605.97
F600	285.71	915.53	895.49
SGL 8x30	909.1		222.96*
AP 4-60	526.3	1104.59	1384.63

In the chart above the carbons F100 and OLC 12x30 were unable to absorb the methylene blue due to small pore sizes. This means that the methylene blue molecule is much larger than the iodine which is better used for carbons with smaller pores. As seen the larger methylene blue and iodine number are determines that the pore size of the carbons will be larger, meaning that the carbon is able to absorb the methylene blue and iodine.

*without iodine number (in preparation)

Conclusion

Overall, this research project was successful in creating a standard method procedure for determining methylene blue numbers. We came up with three different parameters for the optimization of activated carbon. These methods will be used in combination with factorial analysis to optimize preparation of carbons from natural products. Each of these factors, methylene blue number, iodine number, and surface area will be used as response factors to determine optimal conditions for carbon preparation.