Analysis of FTIR spectra of carbon materials: subbituminous coal, graphene oxide, graphene nanoplatelets, and carbon nanotubes

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Subbituminous coal is a major factor of Wyoming's economy. However, cleaner sources of energy, such as solar energy, wind energy, and hydroelectric energy, are highly competitive towards natural gas, oil—and most importantly—coal. Coal is mostly carbon which begs the question, "What carbon materials can be extracted or produced from coal, both efficiently and economically?" to develop new markets for this resource. Discovering a second major purpose for subbituminous coal, aside from its energy uses, would boost Wyoming's economy.

A goal of this research was to establish Fourier transform infrared spectroscopy, or FTIR spectra, of known carbon materials to compare to samples of coal following experimental treatment. The known carbon materials that were tested included carbon nanotubes, graphene nanoplatelet aggregates, and graphene oxide. The subbituminous coal FTIR spectra were then analyzed and compared to spectra obtained from the known carbon materials. Throughout this research, the relationship between mass and thickness of the salt plates was also analyzed. Details of the FTIR spectra comparisons, as well as the observed salt plate relationship, will be reported.

INTRODUCTION

The ultimate goal of this research was to find an efficient and economical method to turn Wyoming's subbituminous coal into graphene. Graphene is a material of the future—conductive and flexible with a high tensile strength. It seems theoretically possible to make graphene out of coal because graphene is a single, 2-D layer of carbon atoms, and coal is mostly carbon.

Ye et al. (2015) described a procedure turning anthracite coal into graphene quantum dots. This research used subbituminous coal to paralleling their procedure because it is the most abundant type of coal in Wyoming.

A review of literature suggested the most common technology to identify graphene or graphene particles was Raman Spectroscopy. However, a Raman Spectrometer was not available throughout this research. Therefore, an alternative identification method was needed to determine if the experimental coal flakes contained graphene. Fourier transform infrared spectroscopy (FTIR) was determined to be the best method for identifying carbon materials given the technology available. However, it was quickly discovered that creating and analyzing carbon materials (carbon nanotubes (multi-walled), graphene nanoplatelet aggregates (sub-micronparticles), and graphene oxide (powder)) via FTIR spectra is difficult.

Due to the dark shades associated with coal and carbon, a very small concentration (ranging from 0.05% to 1.0%) of each of the carbon materials was required to make successful Potassium Bromide, or KBr, pellets. The variability during the creation of the KBr plates also greatly affected the quality of the KBr pellets and the results. Therefore, multiple aspects of the KBr pellet preparation and creation had to be consistently controlled.

METHODS

As described in Ye et al. (2015), the subbituminous coal was ground in a mortar and pestle for roughly 45 seconds. 300.0mg of the ground subbituminous coal were added into a 10mL round-bottom flask. Under the fume hood and using a micropipette, 6.000mL of 18M Sulfuric Acid and 2.000mL of 15M Nitric Acid were added to the flask. The flask was sonicated for two hours, heated, and stirred in a water bath for 18 hours at 75°C. The solution was cooled to room temperature, poured into a 400mL beaker containing 100mL of crushed ice, and titrated with 3M Sodium Hydroxide until a pH of 7 was reached. The neutralized mixture was filtered and dialyzed through 6-8k Da membrane for six days. After purification, the solution was completely dehydrated, and the flask with the remaining coal flakes was sealed using parafilm for future testing.

For FTIR spectra, concentrations ranging from 0.05% to 1.0% of each carbon material in dried Potassium Bromide were massed and combined in a clean mortar and pestle for 30 seconds. KBr pellets were produced in a KBr bolt press using ~0.0750g of sample with bolts tightened to 30 ft. lbs. The pellet was poked out of the press and a digital caliper was used to measure the center thickness of the KBr pellet.

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RESULTS

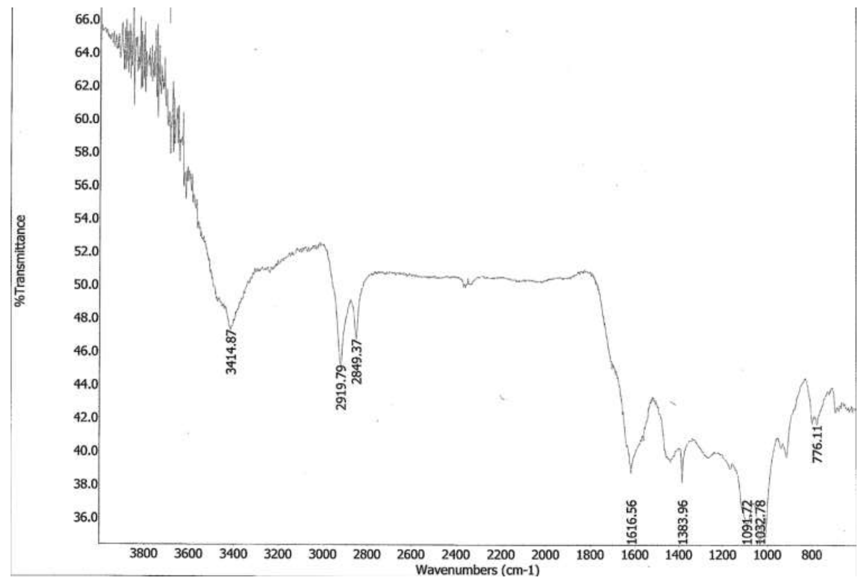

Figure 1: Untreated Coal 1.0%

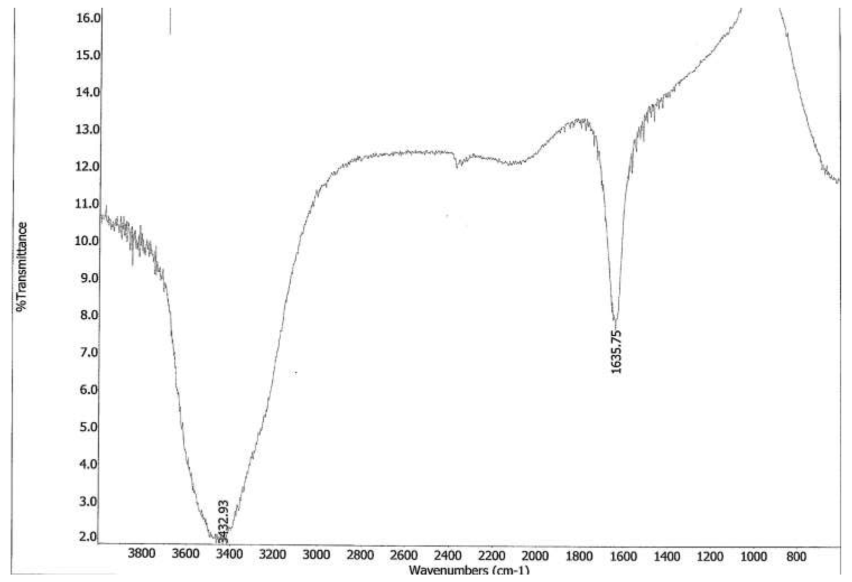

Figure 2: Graphene Nanoplatelets 0.2%

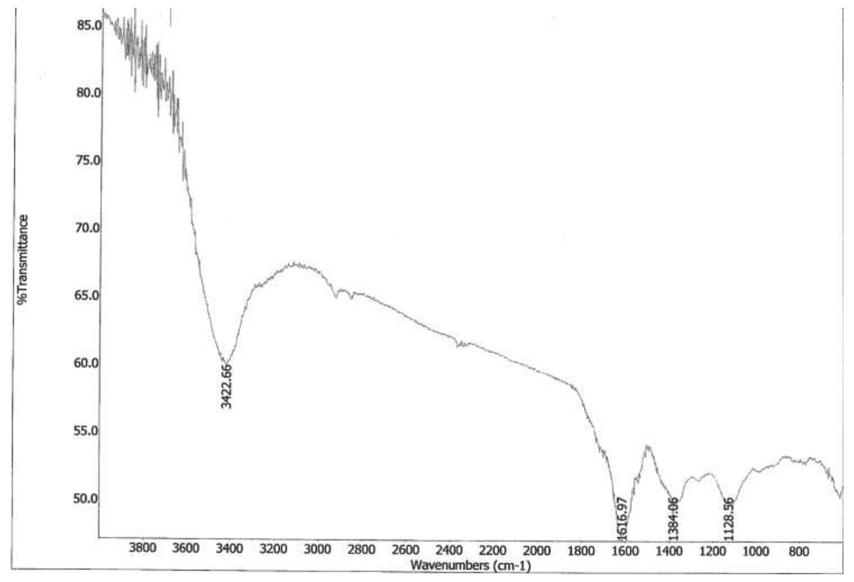

Figure 3: Coal Flakes 0.2%

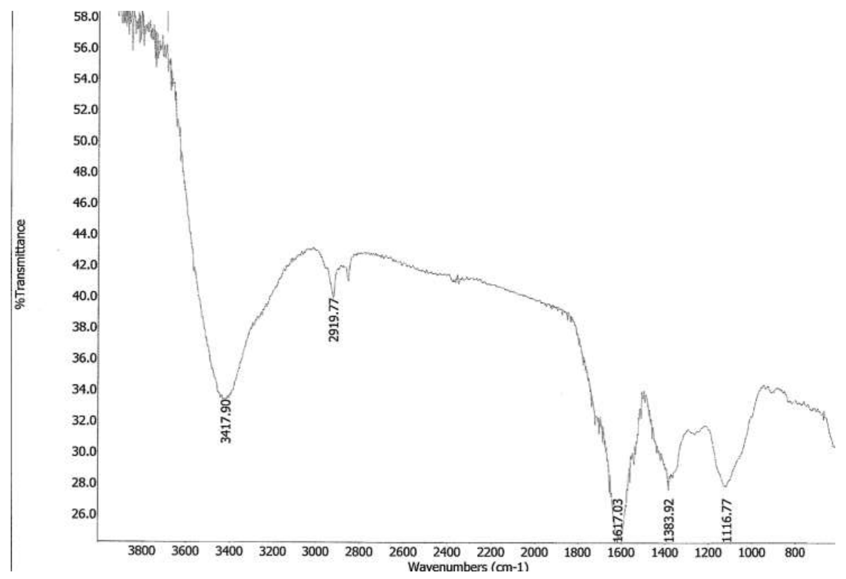

Figure 4: Coal Flakes 0.5%

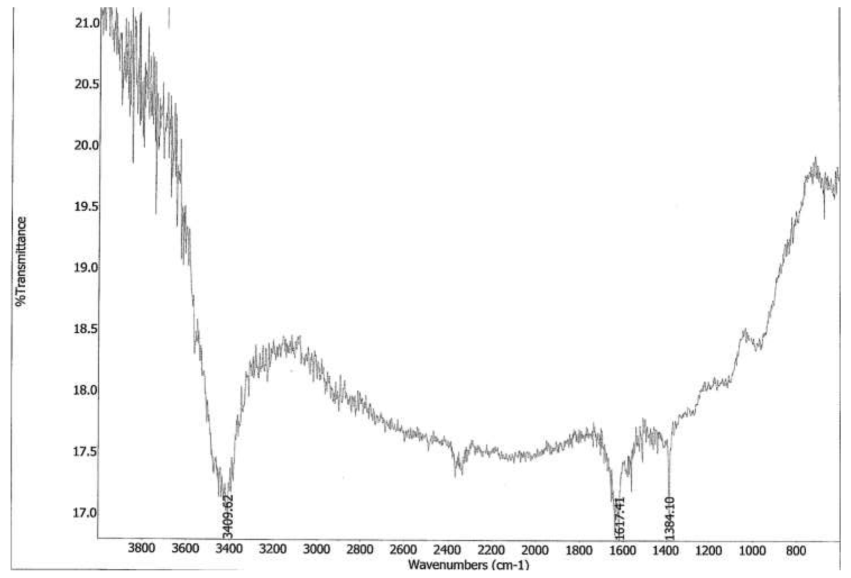

Figure 5: Carbon Nanotubes 0.05%

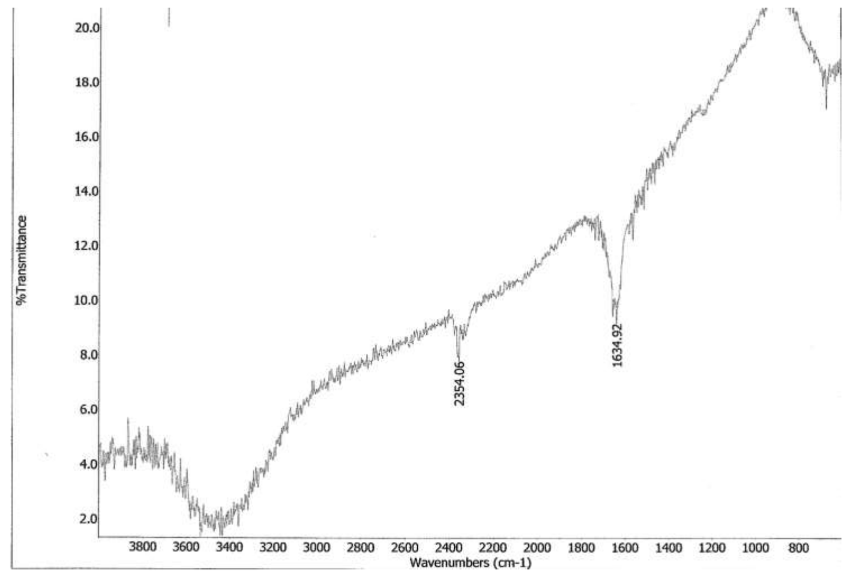

Figure 6: Graphene Oxide 0.3% with 0.05% Graphene Oxide Background

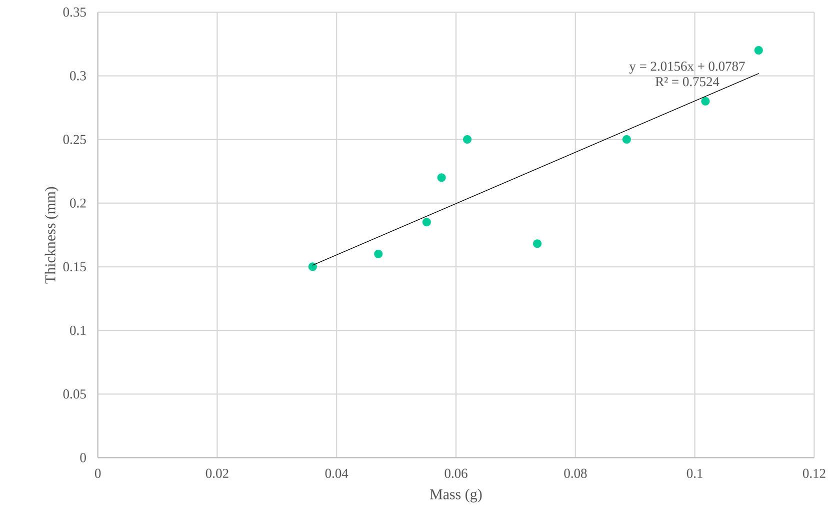

Figure 7: The Effect of Mass on KBr Plate Thickness

Figure 8: Fluorescence of Coal Flakes

DISCUSSION

The experimental coal flakes consistently produced a FTIR spectra with three signal peaks. Both the 0.2% and the 0.5% coal flake concentrations produced peaks at 1617 cm^{-1} , 1384 cm^{-1}, and ~1120 cm^{-1} (see *Figures 3 & 4*). The 1.0% untreated coal's FTIR spectra produced peaks at 1617 cm^{-1}, 1384 cm^{-1}, 1092 cm^{-1}, and 1033 cm^{-1} (see *Figure 1)*. The 0.05% carbon nanotubes' (multi-walled) FTIR spectra produced peaks at 1617 cm^{-1} and 1384 cm^{-1} (see *Figure 5*). The 0.2% graphene nanoplatelet aggregates' (sub-micronparticles) FTIR spectra produced a peak at 1636 cm^{-1} (see *Figure 2*). The 0.3% graphene oxide's (powder) FTIR spectra produced peaks at 2354 cm^{-1} and 1635 cm^{-1} (see *Figure 6*).

The relationship between pellet mass and thickness (see *Figure 7*) was determined to follow the linear equation of: thickness = 2.0156(mass) + 0.0787. This linear relationship had an R^2 value of 0.7524.

The fluorescence of the coal flakes was qualitatively analyzed (see *Figure 8*).

CONCLUSION

The FTIR spectra peaks of the coal flakes did not match the peaks produced in the three known carbon materials' FTIR spectra. The coal flake's peaks most closely matched the untreated coal's peaks with a discrepancy at ~1120 cm^{-1}. The significance of the unique coal flake peak was unable to be analyzed because that ~1120 cm^{-1} peak did not show up on any other carbon materials' FTIR spectra or in any literature. However, the detailed KBr pellet preparation process was determined to be reproducible. To produce the best FTIR spectra, the 0.3% graphene oxide was ran with 0.05% graphene oxide background (see *Figure 6*); All the other FTIR spectra on this poster were ran with a normal air background.

The R^2 value for relationship between pellet mass and thickness (see *Figure* 7) was determined to be 0.7524 which concludes that a linear relationship exists between KBr pellet mass and thickness. This linear relationship is with one average pellet thickness for 44 trials with a mass of ~0.075g.

A green fluorescence emitted from the experimental coal flakes can be seen in *Figure 8*. However, due to technology limitations, the fluorescence observations could not be quantified.

In conclusion, the experimental coal flakes' FTIR spectra did not match any of the known carbon materials' peaks consistently. Due to the different concentrations and variability in the production of KBr pellets, the conclusion of creating graphene out of subbituminous coal is still unknown.

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