



Semicontinuous supercritical extraction
by Stanley Stephen Smith

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in
Chemical Engineering
Montana State University
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Abstract:

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APPROVAL

of a thesis submitted by

Stanley Stephen Smith

This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

August 2, 1983

Date

James P. Scarsik
Chairperson, Graduate Committee

Approved for the Major Department

Aug 2, 1983

Date

John T. Sears
Head, Major Department

Approved for the College of Graduate Studies

22 Aug. 1983

Date

Michael Malone
Graduate Dean

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ABSTRACT

Peat has been viewed as a possible alternate source of energy, and extractions with supercritical gases have been done to utilize such sources. The objective of this investigation is a screening study of the extraction of peat in a semicontinuous extractor with an alkaline methanol solvent in a supercritical state. The extraction column was a 12-inch long, stainless steel, one-inch diameter pipe. The solvent was pumped at the rate of 2.1 to 2.2 milliliters per minute. The solvent in the extract product was removed by a distillation under vacuum, and a moisture and ash analysis was conducted on the residue. A Soxhlet extraction with tetrahydrofuran (THF) was also conducted on some of the residue products; thus, two yields were determined, a total moisture- and ash-free yield and a THF-soluble yield. The screening study entailed the four pressure and temperature combinations possible from 1760 and 2930 psia and 250° and 280° C. Two aqueous experiments were conducted to confirm a possible link between extract yields and pH of the solvent via the water gas-shift reaction. The ash analysis was not appropriate in the determination of yield for residue with large sodium hydroxide:organic content ratios. The sodium hydroxide was also soluble in THF, which rendered problems with that yield determination. Changes in yield due to temperature and changes in yield due to basicity were pressure dependent. Carbon-13 nuclear magnetic resonance spectroscopy was used for characterization of some products. Future studies might include utilization of the extraction apparatus for residence time distribution studies and selective fractionation studies. Carbon monoxide/alkaline systems might also be studied.

INTRODUCTION

Because of a dwindling supply of petroleum resources, the search for alternate sources of energy and chemical feedstocks has been the center of many investigations in the last few decades. Coal has been given a large amount of attention as an alternative, but peat has also been getting some notice.

Many possibilities exist for the utilization of alternative fossil fuel resources. Presently, coal is used primarily as a fuel for power generating facilities. However, liquid hydrocarbons have been produced from coal for nearly two centuries. Various gasification and liquefaction processes exist for coal. Extraction by fluids under supercritical conditions may be a very promising method for recovery of hydrocarbons and related compounds. In the past decade, the National Coal Board in Great Britain has pioneered much of this type of work. Polar and caustic solvents have also been successful solvents for extraction of hydrocarbons.

Characterization of the extractive products is an important step toward utilization of the process under investigation. Techniques are continually improving, and characterization has been greatly simplified. Nuclear magnetic resonance spectroscopy is one method which has proven very useful.

A caustic polar solvent in a supercritical state may be successful in a hydrocarbon extraction. Because of peat's potential as an alternative energy and feedstock source, peat may serve as a suitable substrate.

RESEARCH OBJECTIVE

The main objective of this study was to investigate a supercritical extraction of peat with a basic methanol solvent. The extraction was to be done in a semicontinuous fashion, that is, the solvent would be the mobile phase in the extraction apparatus. Emphasis was to be put on an overall yield of organic material which was extracted from the peat, but some characterization of the products was also of interest.

Specific objectives might best be represented by two major divisions. The first was a screening study which would entail the supercritical extraction of peat with caustic methanol. The investigation was to be centered around two temperatures, two pressures, and three methanol solvents which varied in degree of alkalinity. This would require a method by which inorganic and organic fractions of the product might be distinguished to provide a basis for yield determination.

The second objective was to do fundamental characterization of various product outputs with carbon-13 NMR spectroscopy. The idea was to determine if the technique might be used to distinguish fundamental group differences which were caused by the various parameter and solvent changes.

BACKGROUND AND RELATED RESEARCH

Peat: An Alternative

Utilization of peat for energy is not a novel idea; peat has been used in Europe for centuries. Soper and Osbon {40:1} indicated that during World War I, an extensive survey of peat quantity and quality in the United States was done and possible uses of peat were suggested. Bodle, Punwani and Mensinger {7:559} stated that in the United States alone, some 1400 quadrillion British thermal units of easily recoverable fuel exist as peat. Peat also shows significant potential as a chemical feedstock.

Peat, the partially decayed debris of vegetable matter, is known as a young coal. The coal sequence begins with vegetative matter which is sequentially converted to peat, lignite, bituminous coal, anthracite and graphite (Soper and Osbon {40:7}). Peat is formed when the vegetable matter decays slowly via the regulation of available oxygen. This pertains to vegetation which is submerged in water. The fuel value of peat stems from the large portion of fixed carbon which is retained from the cellulosic structure during the slow decay process (Soper and Osbon {40:6-7}).

Like other fossil fuels, a large diversity exists in peat due to the plant varieties from which the peat had been formed, differences in climate, ages of deposits, water levels, and sediment deposits (Soper and Osbon {40:13}).

Peat is characteristically porous and very wet in its natural environment; however, peat can be air-dried very easily (Bodle, Punwani and Mensinger {7:561}). Peat also is acidic because of the presence of humic acids, the result of bacterial action (Higachi {17:1}). A typical ultimate analysis of peat on a moisture- and ash-free basis would be (Soper and Osbon {40:16}):

59.5	%	Carbon
5.5	%	Hydrogen
33.0	%	Oxygen
2.0	%	Nitrogen
0.2-0.6	%	Sulfur

Peat generally has a low content of sulfur, but the nitrogen content is generally high.

Soper and Osbon {40:17} indicated that ash can range from three to 30 percent of the dry weight of the peat and is traceable either to the plant cells or to mineral matter. Myklebust {23:8-9} indicated that the straight combustion of peat as a fuel has the same problems as the burning of coal; such problems are sulfur and nitrogen emissions and fly ash. But peat does have prospects of becoming a significant alternative to petroleum.

Extraction via Supercritical Fluids

Supercritical fluids. According to Smith and Van Ness {37:57-58}, homogeneous fluids are normally divided into two classes, liquids and gases, but the two phases become

indistinguishable at what is called the critical point. The vaporization curve of a pure material on a pressure versus temperature diagram terminates at the critical point, which has the coordinates of the critical pressure and critical temperature. These represent the highest temperature and pressure at which a pure material can exist in vapor-liquid equilibrium. Gangoli and Thodos {11:209} defined a supercritical gas as one which cannot be condensed to a liquid by an increase in pressure, that is, the temperature is above the critical temperature.

People in chromatographic sciences realized the benefits that a supercritical gas provided. Ample literature was available in this field about the characteristics of supercritical gases. Peaden et al. {27:1070} and Gere, Board and McMangill {12:736} stated some supercritical gas properties and characteristics, and they presented some advantages of the idea as it pertained to chromatography. Gere, Board and McMangill also investigated what effects a packed column had on supercritical gas properties. Such observations included: (1) as the mobile phase approaches the critical point, the density of the gas approaches that of a liquid; (2) solute diffusion coefficients are approximately two orders of magnitude more than those found in liquids; and (3) the fluid has a viscosity approximately that of gas and two orders of magnitude less than that of the corresponding liquid.

The characteristics that make a supercritical fluid an attractive solvent for chromatography are also attractive from an extraction standpoint. Williams {44:1769} summarized some advantages: (1) supercritical fluid extraction is particularly effective for the isolation of substances of medium molecular weight and relatively low polarity; (2) the separation can be accomplished at moderate temperatures and consequently be applied to the recovery of heat-labile substances of low volatility; (3) a supercritical fluid has high diffusivity and low viscosity, thus allowing rapid extraction and phase separation; (4) the solvent power can be varied over a wide range by varying pressure and temperature; and (5) the fluid is easily recovered from the extract and from extraction residue as a result of its high volatility.

An extraction with a supercritical fluid involves both an increase in vapor pressure and a phase separation, thus Williams {44:1769} pointed out the technique is related to distillation and extraction with liquid solvents. Work of Paul and Wise {26} offered a comprehensive review of the principles of extraction with compressed gases, and the relation to distillation and liquid-liquid extraction was discussed on a physical basis.

Paul and Wise {26:21-25} suggested that the solvent should have a critical temperature near the desired extraction temperature. This temperature should be such that the

solute is relatively volatile at the desired extraction pressure, which should be as high as possible.

Williams {44:1770-1772} also presented some theory of supercritical gas extraction with thermodynamic arguments, but with limited success. The availability of high-speed computers and the continuing effort which is being applied to thermodynamic analysis might lead to a better understanding of the dense fluid state and thus to the improvement of state equations and identification of new applications for supercritical gas extraction. He utilized experimental data to illustrate general principles and he classified phase behavior of some binary and ternary systems; a table was used to summarize the systems. Applications of supercritical extractions in various industries were also included.

Worthy {45} presented a general overview of the prospects of supercritical fluids. Gangoli and Thodos {11} indicated some properties and advantages of a supercritical gas along with some history about supercritical gas. They presented applications and compared liquid-liquid extraction to that of a supercritical gas extraction. Thermodynamic theory of supercritical extractions was also discussed.

Gangoli and Thodos {11:214-215} suggested a sequence of events which may occur during an extraction with a supercritical gas. The process sequence may be broken down into five steps: (1) the supercritical gas penetrates the micro-pore structure of the substrate; (2) the large molecular

aggregates are depolymerized and the resulting products dissolve in the solvent; (3) molecular bonds between the molecular species which are to be extracted and the substrate are broken; (4) the extract and the solvent gas diffuse from the micropore structure; and (5) the extract is recovered from the solvent gas as a precipitate or condensate by a reduction in pressure. They also proposed that the mechanism of gas flow through the substrate is a combination of Knudsen diffusion through the small pores and bulk diffusion through large pores, or permeation through the fracture system of the substrate.

Maddocks, Gibson and Williams {19} brought forth key points of a supercritical extraction process which included a view of process parameters such as solvent gas and coal type. They presented process economics and a flow model of a simplified, commercial scale supercritical extraction process. Results were listed which pertained to extraction of coal with toluene and paracresol.

Bott {8:232} also presented some of the economics of supercritical extraction processes. He pointed out that supercritical extraction techniques are high pressure processes which require special equipment. Unless the process includes energy recovery steps or maintains a low energy demand in some way, the technique might only be economical for high value products. Patents of various processes from a number of countries were listed. Some data on the

supercritical fluid extraction of tar sands and peat with n-pentane and benzene were given, as was information on supercritical carbon dioxide.

Scarrah {29} and Myklebust {23} both worked with supercritical extractions on peat in a batch extraction apparatus. The research was geared toward solvent behavior, and some solvents which were used were water, methanol and acetone. Scarrah ({30}, {31} and {32}) utilized a semicontinuous extraction apparatus and investigated the possibility of supercritical gas extractions on lignite and peat with various solvent systems and pressure fractionation.

Semicontinuous extraction. Most of the previous supercritical extraction work has been done in batch reactors. However, Scarrah {30:11} has done some work with the semicontinuous extraction of lignite with supercritical fluids, and he has discussed some of the advantages and disadvantages with the semicontinuous extraction apparatus. The pressure is easy to control, especially when compared to a closed batch system. The saturation of the solvent with solute is not a problem because pure solvent is always supplied in a continuous extraction. The last major advantage is the ease of product collection.

Some disadvantages of the semicontinuous method include the need for continual attention during an experiment, long experimental run times, and the increase in components which increase the chances for malfunctions (Scarrah {30:11}).

Another disadvantage is a plugging problem with some substrates and solvents.

Polar and alkaline solvents. For the success of an extraction process, the choice of solvent is significant. Various factors which make one solvent different from another include the ability to accept protons or contribute protons and dipolar characteristics.

Gangoli and Thodos {11:215} noted that polar solvents exert a stronger dissociating or depolymerizing action on coal than non-polar solvents in a supercritical state. Both water and methanol are polar in nature and both are relatively inexpensive. The major difference is that methanol is a strong proton-acceptor and a weak proton-donor but water is a strong proton-donor and weak proton-acceptor (Snyder {39:229}).

Methanol-water systems have been noted to extract up to 90 percent of the organics in oil shale at supercritical conditions {24:54}. The methanol was thought to be the major organic selective reagent. Scarrah {29} also suggested that methanol might be a good potential solvent for supercritical extractions.

Alkaline aqueous solvents have been successfully used to increase yields in hydrocarbon type extractions. Ross et al. {28} indicated that an increase in yield of benzene-soluble products from coal extractions is possible if the basicity of the aqueous solvent is increased. Basic

solvents have also been used to separate humic acids from coal (Higachi {17:1}).

The latter results indicate that a caustic polar solvent in a supercritical state may be successful in a hydrocarbon extraction. Because of its acidic nature, peat may serve well as a substrate in such investigations.

Characterization and Identification

Separations. A conglomerate of chemical species evolve from any type of effort which is geared toward the removal of hydrocarbons from fossil type resources. Uden et al. {43:38} suggested that the chemical species in such products may form the basis of major petrochemical feedstocks if characterization and separation can be achieved. They discussed the importance of characterization of hydrocarbon products, and they suggested a method for the separation of tar acid and tar base fractions of shale oil.

Farcasiu, Mitchell and Whitehurst {10} discussed a method for fractionation and characterization of Solvent Refined Coal through solvent extraction. Three major divisions exist. The oils are the hexane solubles, the asphaltenes are the hexane insoluble-benzene soluble compounds, and the less soluble materials are benzene insoluble but soluble in pyridine. Sternberg, Raymond and Schweighardt {41:49} reported that the asphaltene fractions have generally an acid-base structure which can also be divided into the

two respective components. The hydrogen-bonded structure of the asphaltenes is compatible with their solubility characteristics. Thus, the characteristics of a solute are very dependent upon the solvent. Thin-layer chromatography, with the aid of visualization sprays, has been used to confirm the presence of aromatic phenols in the acidic component of petroleum asphaltenes. Mass spectrometry has been a very useful method for characterization studies (Ozubko, Clugston and Furimsky {25}). Nuclear magnetic resonance (NMR) spectroscopy, especially carbon-13 NMR, has also become very important in characterization studies.

Bartle, Martin and Williams {4} recognized the need for characterization of extraction products and reported on their work on a supercritical extraction of coal. The solvent was toluene. The characterization began with physical separations which utilized various solvents in a gel-permeation column. Work with NMR spectroscopy was also presented. The work of Bartle et al. {3} exemplified structural analysis of supercritical gas extracts of coal with emphasis on NMR spectroscopy.

NMR spectroscopy. Proton NMR spectroscopy can be used to distinguish the various environments which are present in the test sample, and thus provide an idea of aromatic versus aliphatic portions of the sample. When coupled with an ultimate analysis, the various components are often revealed. Carbon-13 NMR spectroscopy has become useful in that it

reveals direct structural information about the carbon groups which are present. This is especially useful for detection of those groups which do not contain hydrogen such as internal aromatic carbon, carbonyl, and aromatic ether. The advent of pulsed Fourier transform NMR and proton decoupling has made possible the distinction of well-resolved bands in both aromatic and aliphatic regions of the spectra of various coal derivatives (Snape, Ladner and Bartle {38:2189}).

Because of the distinct separation of aliphatic and aromatic carbon signals on a carbon-13 spectrum, when special precautions are taken, direct integration provides a ratio of aromatic to aliphatic carbon (Abraham and Loftus {2:150-160}, Sklenar et al. {35}, and Maekawa, Yoshida and Yoshida {20}). This has advantages over chromatographic-gravimetric methods for characterization of various compounds like aryl compounds with alkyl side chains. The physical separation of the aliphatic and aromatic carbon is impossible, and all the carbon of the compound is put in the aromatic or saturate fraction (Shoolery and Budde {34:1461}). However, any prior physical separation of a complex hydrocarbon mixture aids in the identification of groups or compounds.

Abraham and Loftus {2} presented a good background for the basic theory and terminology of NMR spectroscopy. Pulsed Fourier transform spectroscopy was also discussed.

The basis of NMR spectroscopy for identification of specific groups in different environments (shielding effects) is the chemical shift. A chemical shift is the response of various groups to radio frequencies in a magnetic field (Morrison and Boyd {22:419-420}). The spectrum is composed of signals which represent chemical shifts of various nuclei interactions. The reference point from which chemical shifts are measured is generally from an actual compound. Tetramethylsilane is a common reference compound and has been assigned the value of 0.0 part per million (ppm) on the delta scale (Abraham and Loftus {2:13-14}). This is on the right of the spectrum and most chemical shift signals occur downfield or to the left of 0.0 ppm.

Deuteriochloroform is often used as a solvent and reference because its signal at 77 ppm does not interfere with either the aliphatic or aromatic regions of the spectra field. The deuteriochloroform also provides the lock signal which is required for pulsed Fourier transform NMR spectroscopy. The lock signal merely keeps the radio frequency in synchronization with the inevitable small variations of the applied magnetic field (Abraham and Loftus {2:8}).

The most obvious division in a carbon-13 spectrum was that between aliphatic and aromatic carbon. Aliphatic carbon were represented in the 10 to 50 ppm range, and aromatic carbon were generally located downfield of 120 ppm. Snape

Ladner and Bartle {38:2189} suggested that solvent, concentration, and paramagnetic effects should not be forgotten because variations of up to 0.5 ppm had been observed in various situations.

Maekawa, Yoshida and Yoshida {20:864} stated that specific isolation of each individual compound was clearly impossible; however, some individual compound assignments might be done. Recent work with NMR spectroscopy was done by Coleman and Boyd {9} whereby the silicon-29 nucleus was used to determine hydroxyl-containing compounds in shale oil crude. Shelley and Munk {33} pointed out the availability of library searches for computer prediction of substructures from carbon-13 spectra. Carbon-13 enrichment procedures were used by Mikita, Steelink and Wershaw {21} for the determination of hydroxyl functionality in humic substances. Various chemical shifts which were common in humic substances were assigned.

The energy crisis has encouraged much research to be done with supercritical extraction and has focused some attention on peat as an alternative. The theory, characteristics, and applications of supercritical gas extractions are being developed. Characterization methods, especially those associated with NMR spectroscopy, are proving to be powerful tools in the search for alternatives.

RESEARCH DESIGN AND PROCEDURE

Experimental Design

The following approach towards the objective of this investigation was taken. Two water runs were planned to be conducted to confirm previous work by Ross et al. {28}, that is, an increase in alkalinity will increase the yield in an aqueous system. These experiments were to be done at 400°C and at a pressure of 3030 psia. One experiment would utilize pure water; the other experiment would employ a 0.5 molar sodium hydroxide solution.

The first objective, the screening study, was to be achieved with four pressure-temperature (P-T) combinations which are indicated in Table 1. A screening was to be done which involved three different methanol solvents of various degrees of basicity. Yield was to be based on the moisture- and ash-free (MAF) content of the peat. To

Table 1. Experimental Pressures and Temperatures.

Combination	1	2	3	4
Pressure (psia)	1760	1760	2930	2930
Temperature (° C)	250	280	250	280

psia - pounds per square inch absolute

° C - degrees centigrade

