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IN-SITU GENERATION OF HYDROCARBONS FROM KEROGEN USING ELECTROMAGNETIC RADIATION

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IN-SITU GENERATION OF HYDROCARBONS FROM KEROGEN USING
ELECTROMAGNETIC RADIATION

by
Forrest Green

A thesis submitted in partial fulfillment of the
requirements for the degree of

Master of Science in Petroleum Engineering

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Abstract

From using surface seeps to modern day exploitation of hydrocarbon source rocks, the petroleum industry is awaiting the next big innovation, accelerated kerogen conversion. In the industry's history, electromagnetic radiation (EMR) has been used for exploration and now it will be taken to the extreme by determining an effective and efficient method to synthetically generate readily available hydrocarbons. The study starts with examining and defining what kerogen is and its importance as a source material. A strong understanding of what kerogen is, and an analysis of other in-situ projects presents an opportunity to study the finer details of kerogen's conversion to hydrocarbons. In-situ projects from around the world were studied to eliminate unsuccessful methods of conversion. The unsuccessful methods highlight key details that are needed for developing a successful conversion methodology. To understand the method being developed, it was necessary to build a strong understanding regarding the process of thermal maturation. For this study, samples rich in organic matter were tested in various methods of EMR exposure. Bakken formation samples were used because of their high organic concentrations and proximity to the research center. This study additionally focuses on microwaves, a region of larger waves in the electromagnetic spectrum and their impact on kerogen. Methods of exposure include using dry cores, water-soaked cores, and cores submerged in water baths. Unaltered Bakken cores and water-soaked cores exposed to the microwaves showed better results of hydrocarbon generation than those tested in a water bath. The effectiveness of EMR kerogen conversion was compared to the results of a simple retort analysis conducted within the expected thermal maturation temperature range for 10-hours. Microwave tests were conducted in the same temperature range with evidence of successful hydrocarbon generation. An estimate of the energy requirement for kerogen conversion with EMR was analyzed and the inaccuracy of this requirement value was discussed. Experiments conducted in this study showed that microwaves can be used to generate hydrocarbon fluids. Conclusions of the study acknowledge that the EMR methodology of kerogen conversion is possible but further experimentation is required.

Keywords: kerogen, thermal maturation, kerogen conversion, hydrocarbon generation, Bakken, electromagnetic radiation, microwaves

Dedication

I wish to dedicate this my family, past and present. Thank you to my parents, who always believed in me and my endeavors. To my grandmother, who unfortunately passed early in my college career, you implanted the Montana lifestyle on me that I have grown to live and love so much during my time here at Montana Tech. Finally, my dog, whom I lost at the start of this project. You were always right beside me during this project. You all pushed me to be the best version of myself and work my hardest on whatever I set my mind to.

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Secondly, I would like to thank and commend my advisor, Dr. B. Todd Hoffman. He approached me with the concept and funding for this project. Without his guidance and support, I would have been lost during the project development and conclusion. Through this intellectually intensive process, your guidance and acknowledgments of my concerns pushed this project to completion.

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Lastly, I would like to thank my peers for supporting me in the low spots of this project by providing me with distractions to remind me of the joy that can be experienced while still in school. The last few months of this project seemed to be a never-ending to-do list that you provided me with motivation and pushed me through.

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1. Introduction

In the current production of hydrocarbons reservoirs, a large quantity of oil, gas, and organic matter is left in the reservoir due to the conditions of the formation, the properties of the fluids, and the inability to recover with current technology. This project focuses on kerogen, the organic matter residing in the reservoir. The goal of this project is to determine if microwaves can heat the kerogen and convert it to producible hydrocarbons. If this process proves successful, the kerogen that is left in place in the reservoir could be exposed to microwaves; then, the generated fluids would be extracted through conventional methods of production.

1.1. Kerogen and Hydrocarbon Formation

Kerogen is sedimentary organic matter (OM) that is created by living organic matter and the product of its metabolism. The definition of kerogen is broad and can vary. Some researchers classify kerogen as any deposited OM including soils and slightly buried matter, and others classify it as only compacted and deeply buried (>100 ft) OM under anerobic conditions. In this study, the OM of concern is that which is compacted, solidified, and contained within shale.

Component structures of this OM (carbohydrates, proteins, lipids, and lignans) go through various degrees of decomposition upon burial (Durand, n.d.). This decomposition is called diagenesis, where the physical and chemical structure of the OM is altered at a relatively low temperature. As geologic time progresses, the OM is buried by more sediment increasing temperature and pressure. A visual representation of this burial process is shown in Figure 1.

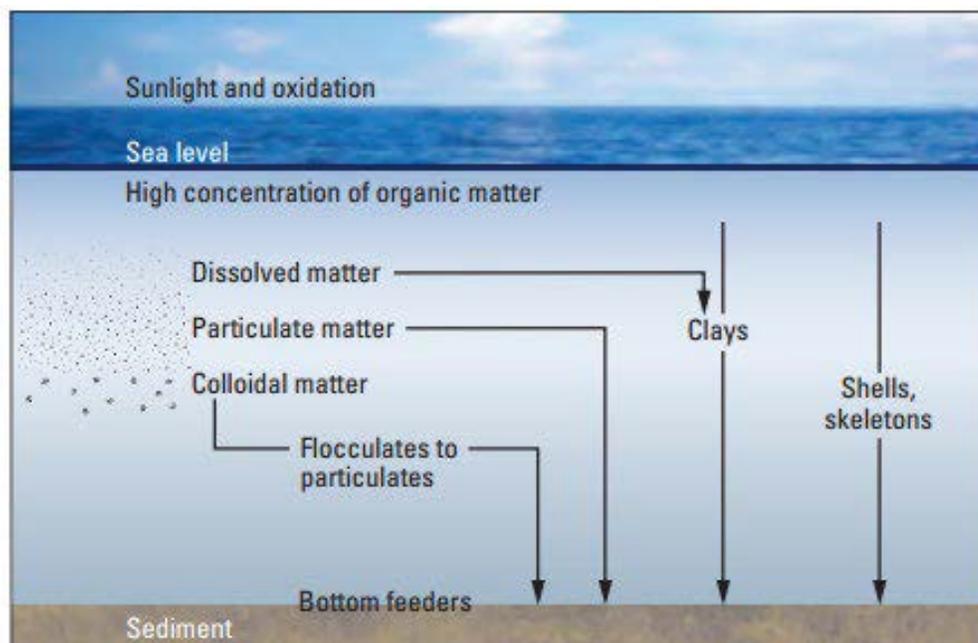


Figure 1: Visual representation of sediment and organic matter deposition for formation of kerogen (McCarthy et al., 2011).

The three types of deposits that result in kerogen are:

1. Lacustrine, deposits from lakes, known as Type 1;
2. Marine, deposits from seas and oceanic environments, known as Type 2; and,
3. Land plants, deposits from swamps, known as Type 3.

Post diagenesis, kerogen begins a process of thermal maturation. This process chemically alters the kerogen into products such as oil, gas, and bitumens. These three products are a result of the burial temperature, pressure, and the chemical composition of the kerogen. To generate oil, the thermal maturation process must maintain a temperature between 100 and 150°C (Allix et al., 2010) within a pressure window. The green oil region in Figure 2 shows as pressure increases, the temperature required to generate oil decreases. At higher temperatures, 150-200°C, more gaseous compounds are developed, such as methane and dry gas. In these higher temperatures some nonhydrocarbon gases, carbon dioxide, nitrogen, and hydrogen sulfide, are produced (McCarthy et al., 2011).

Determining the potential of a kerogen formation to generate hydrocarbons is crucial. One tool used is the measurement of vitrinite reflectance or %Ro, Figure 2. This value can be used to determine the quantity and quality of the fluid type the kerogen can generate. The %Ro value is an average measurement of incident light reflected from the surface of a vitrinite particle contained in a sedimentary rock. Although this process is not exact as the results are dependent on the chemistry of the vitrinite particles, it still allows for an estimate on the product generated from the kerogen.

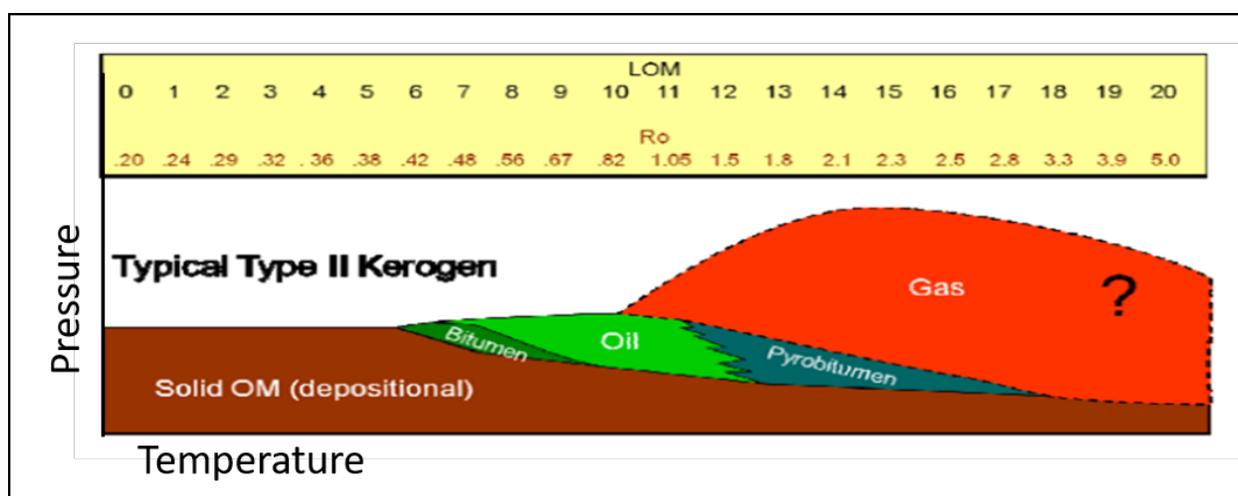


Figure 2: Vitrinite reflectance scale (Barker, 2012).

The maturity of kerogen is used to determine its hydrocarbon generation potential. It is important to remember that not all kerogen will generate hydrocarbons. The hydrocarbon generation potential of a kerogen deposit is determined by the amount of OM remaining after any volume of hydrocarbons has been generated. Kerogen consists of two types of OM, generative material, and non-generative material. As the maturity of kerogen increases, more hydrocarbons are generated, the generative material decreases and non-generative material increases. A young kerogen deposit would be one that has yet to or just started generating hydrocarbons and has a high generative material value. Given the time, the deposit would generate an economically positive volume and be worth producing. Whereas a mature kerogen

deposit would be one that has already generated most of its hydrocarbons, has a lower GOC value (Bondarenko, 2018, p. 178).

1.2. Unconventional Reservoirs

An unconventional reservoir (UR) is a kerogen bearing formation that contains non-migrated hydrocarbons. Some common conditions of these reservoirs are low permeability, high pressures, and high temperatures. The exploitation of URs can be explained through the progression of technology. These reservoirs were deemed poor because vertical wells did not have a large enough drainage area to effectively produce the available hydrocarbons, shown in Figure 3. With the introduction of directional drilling, a much larger area of the reservoir was able to be produced. Then, the addition of multistage hydraulic fracturing made URs more productive because of the possibility to create high permeability zones in low permeability rock. Directional drilling and hydraulic fracturing methods have advanced enough in the last 20-years that they have become the leading production style in North America.

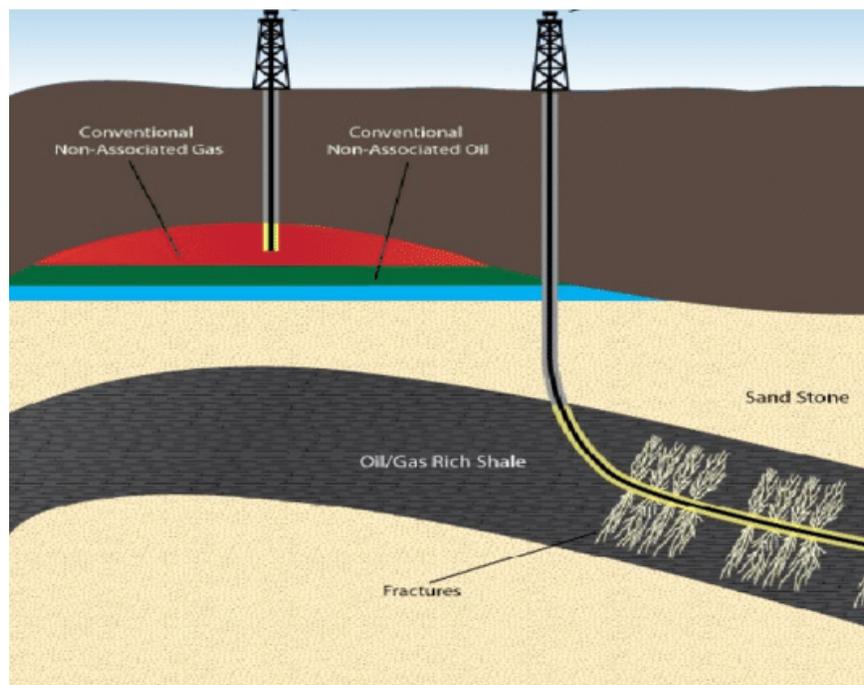


Figure 3: Vertical versus directional drilling technology (Bjørnland et al., 2021).

Current petroleum industry operations are exploiting these high quality and high volumes of hydrocarbons in known URs. Targeted shale formations are commonly comprised of fine sediments, clay, and kerogen. Different formations have varying organic matter quantities. For example, the Upper Bakken (North Dakota, Montana, and Saskatchewan region) formation has an organic matter content of 11% by weight whereas the Wolfcamp formation in the Permian Basin (New Mexico and Texas region) has an organic matter content of 5.4% by weight (Baskin, 1997).

The petroleum industry associates some shales as the source rock of hydrocarbons, the rock where hydrocarbons are created and migrate from. Many hydrocarbon bearing shales exist in the Earth. Some large shale formations in the United States are the Bakken, Marcellus, and Green River shales. Due to the small particle size of the sediments making up shale, the porosity is very low. Often, zones of greater porosity are found in the kerogen particles. In this style of porosity, the storage and movement of oil and gas is controlled by the kerogen.

The middle section of the Bakken is the common target for production using long horizontal wells. This section has a higher porosity and permeability than the upper and lower shale formations. Comparatively, the middle section has a much lower kerogen making it less desirable for a conversion project.

In this project, Bakken shale was used as the research subject. The Bakken shale was selected due to its high kerogen to mass ratio, close location to research center, and availability of testing materials. The Bakken is comprised of three sections, the upper shale, middle silty-dolostone, and lower shale as shown in Figure 4. The entirety is split into these sections (upper,

middle, and lower), based on hydrocarbon content. For this project, three samples from the upper and three samples from the lower Bakken were selected due to their high quantity of kerogen.

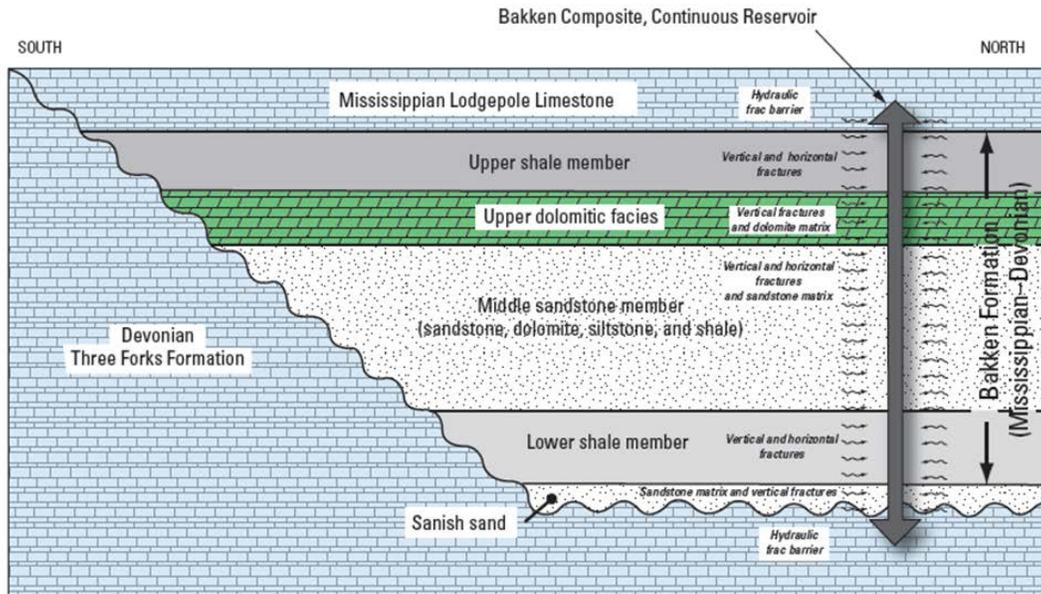


Figure 4: Bakken Formation schematic highlighting three divisions (Patel, 2014).

1.3. Microwaves

Other than a term for a cooking device in most kitchens, a microwave is a type of electromagnetic radiation. Electromagnetic radiation (EMR) is a matter disruption wave traveling at the speed of light that causes a disturbance of the electric and magnetic fields of particles. Waves operate on a sinusoidal pattern, meaning they have a crest and a trough, shown in Figure 5. As the waves move, magnetic and electrical fields of an object in contact with the wave are manipulated.

A wavelength is the measurement between crests of the wave and is measured in units of length. The frequency of a wave is defined as the number of peaks that pass a fixed point in a given time period with a unit of a hertz (Hz). When broken down, the unit of a hertz is the

number of crest passes per second. If 15 wave peaks passed a point in 60 seconds, then the frequency would be 0.25 Hz.

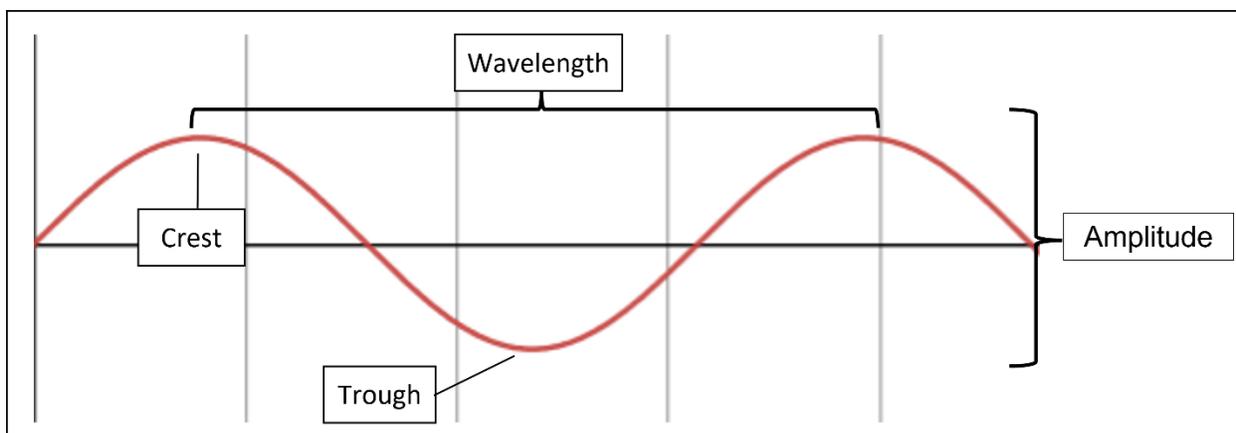


Figure 5: Construction and terminology of a wave.

Electromagnetic radiation exists on a large spectrum, shown in Figure 6, with the largest waves being long radio waves and the smallest waves being gamma rays. Within this spectrum exists the small section of EMR visible to the human eye, visible light waves. Electromagnetic (EM) waves are constructed of an electrical field and a magnetic field that are perpendicular to each other. The waves then propagate away from the source, orthogonal to the electric and magnetic fields. Unlike mechanical waves, EM waves do not need a medium to move through, meaning they can travel through non-reflective mediums and a vacuum. An example of an EM reflective material is aluminum. Aluminum has a high refractive value causing the wave to be reflected with minimal energy loss. This ability of an EM wave to travel through non-reflective material may prove beneficial for reservoir penetration so long as the wave can maintain its energy to the desired depth.

Types of Electromagnetic Waves

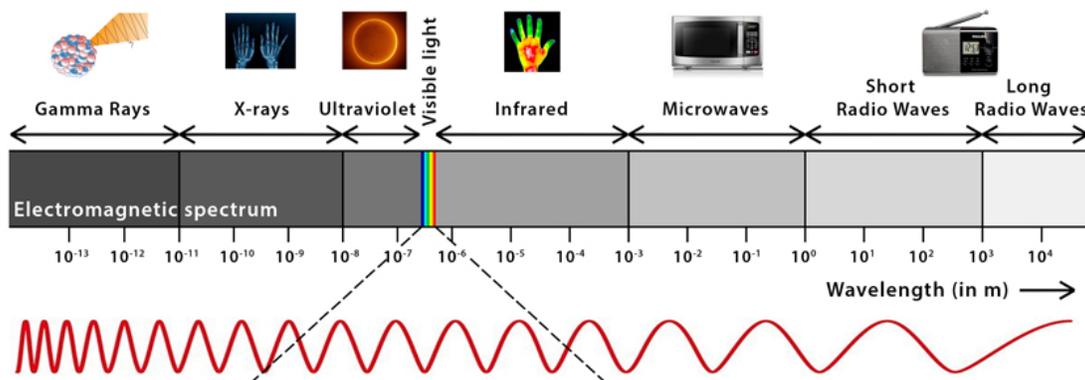


Figure 6: Electromagnetic spectrum and types of waves demonstrating wavelength and frequency (Bhuyan & Mukherjee, 2020).

The term microwaves, covers a large spectrum of wavelengths ranging from 0.001 to 0.3 meters. Standard kitchen microwaves operate with a frequency of 2.45 GHz and a wavelength of 12.2 cm. In a microwave appliance, microwaves are generated in a magnetron, Figure 7, then fed into the cooking chamber through a waveguide. The magnetron is constructed of a single cathode cylinder at the center with a hollow circular anode surrounding it. In the anode are several cavities that are designed to resonate at the desired frequency, 2.45 GHz. A voltage is then applied to the space between the electrodes, typically several kilovolts, creating an electric field. On both sides of this construction are magnets. These magnets create a magnetic field inside the apparatus perpendicular to the electric field. As power is applied, electrons are ejected by the cathode and accelerate rapidly. Due to the magnetic field, these electrons begin to move in a cycloidal path (Vollmer, 2004).

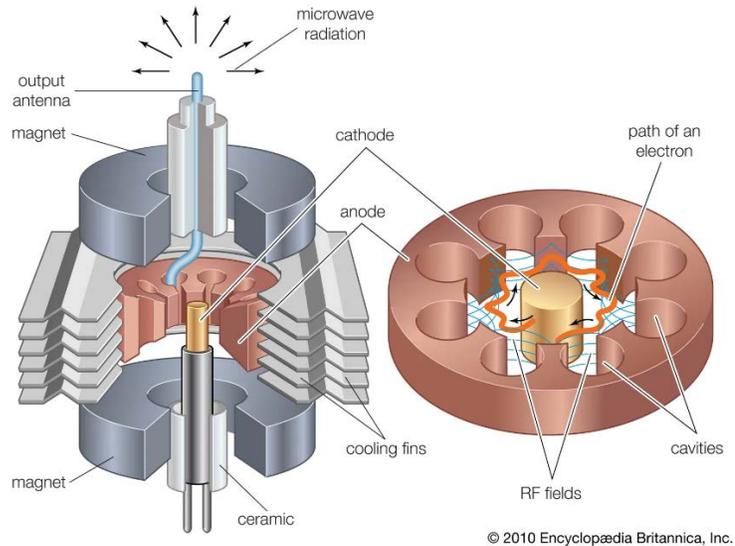


Figure 7: Parts of a magnetron. General construction and layout (Britannica, n.d.).

The chamber is lined with metallic walls creating a Faraday cage so that the waves cannot escape and are reflected around the chamber, demonstrated in Figure 8. The access door, glass door, and light pocket are areas for waves to possibly escape. This escape chance is minimized by placing a metal mesh over them. The holes in the mesh have a smaller diameter than the wavelength of the microwave which allows them to complete the Faraday cage (Vollmer, 2004).

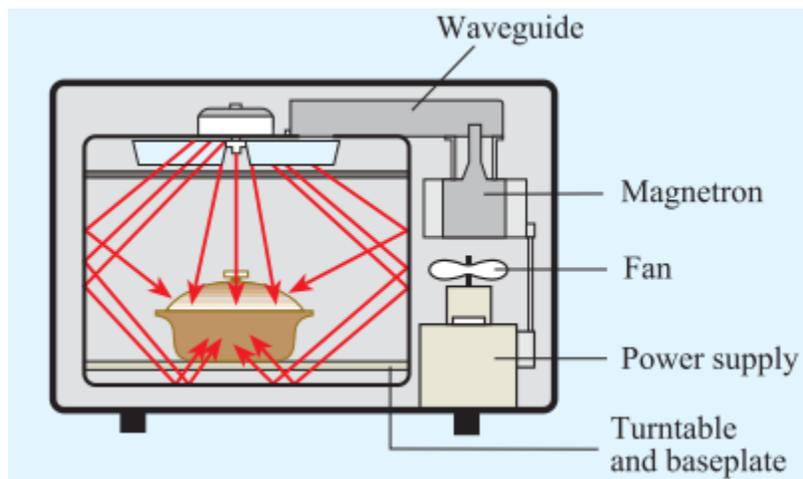


Figure 8: Basic construction of a microwave appliance including magnetron, waveguide, and cooking chamber (Vollmer, 2004).

Electromagnetic waves are polarized which causes them to manipulate the direction of polar particles. As the waves bounce around in the appliance the molecular polarity aligns to match the polar direction of the EM wave (Vollmer, 2004). An example of this molecular motion is detailed in Figure 9. Defined simply, the polar side of the molecule aligns itself to face the peak of the wave. This motion is easily done when the molecules are exposed to lower frequency electric fields. This process is diminished the higher the frequency, 1-10 THz for example (Vollmer, 2004).

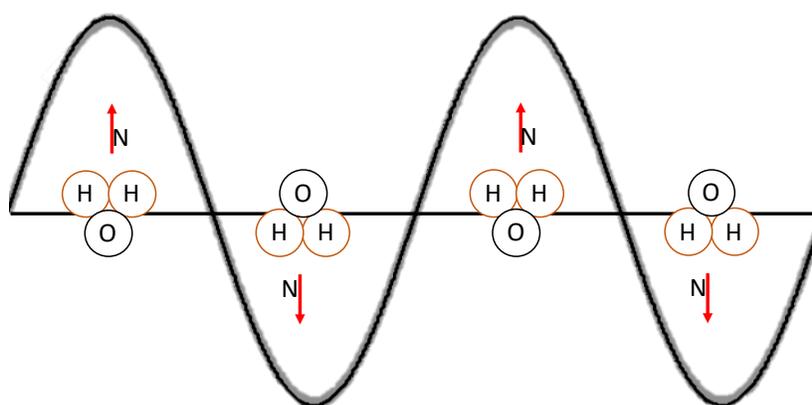


Figure 9: Molecule polarity change through an EMR wave.

As the molecules move to align themselves, the energy of the system increases. As there is a lag in the molecular orientation, power is absorbed from the electric field known as dielectric loss. The energy change of the system is determined by the volume of the object being exposed, the area of the electric field within the system, and the systems dielectric constant (Vollmer, 2004). This would lead to the assumption that with an increase of energy entering the system, the temperature should increase, thus initiation the thermal maturation process.

1.4. Importance of Study

The production possibility out of URs is many orders of magnitude greater than that of conventional reservoirs. This large production potential is due to the kerogen, which is the source of hydrocarbons. Of all the hydrocarbons produced from unconventional reservoirs, significantly more unconverted kerogen exists. It is the scope of this study to test one range of EMR on the conversion process of kerogen into producible hydrocarbons. More specifically, this study aims to develop an understanding of microwaves, their effect on kerogen, and the possibility of kerogen conversion with microwaves. The access to these untapped hydrocarbons would be a tremendous leap into the production possibilities of oil and gas from unconventional reservoirs.

To better define the scope of this project, the following study questions were developed to provide a base for future research and a direction to look.

1. Can kerogen be converted to hydrocarbons using EMR, specifically at the microwave frequency?
2. Can the generated hydrocarbons be collected? If so, what collection device will be effective for obtaining and measuring generated hydrocarbon fluids?
3. How much energy is required per unit of kerogen for conversion to hydrocarbons?
4. How does EMR affect hydrocarbons (existing or generated)?

As this concept of in-situ generation is new to the industry, many questions exist, and small discoveries need to be made to determine a path forward. For now, using these study questions, a direct path of research is set and easily traceable.

2. Relevant Literature

2.1. Industrial History

The usage of petroleum products has been woven into the history of humankind for many thousands of years. Ancient civilizations would worship sacred fires fueled by natural gas surface seeps. As early as 6000 B.C., sailing civilizations used viscous petroleum to waterproof their boats and heat their homes. In Egyptian history, asphalt was used as an embalming mechanism for mummifying the dead (Fagan, 1991).

The Society of Petroleum Engineers (SPE) maintains a technological timeline as shown in Figure 10. According to the SPE, on July 14, 1848, the first oil well was drilled Major Aleveev in Baku, Azerbaijan using a cable drilling system. Eleven years later, August 27, 1859, Colonel Edwin L. Drake and George Bissell discovered oil in the United States (US) when they drilled a 69 foot well in Titusville, Pennsylvania with a homemade rig (Yergin, 1991, p. 27). Due to the newly available fuel source and evolution of industrial technology, the Texas oil boom began in 1901 with a deep gushing oil well near Beaumont, Texas at Spindletop. Using water-based drilling mud, a steam powered drill rig, and fishtail bits, Captain Anthony F. Lucas showed the world what real oil production was with the Lucas 1 well producing “as much as seventy-five thousand barrels per day” (Yergin, 1991, p. 85).

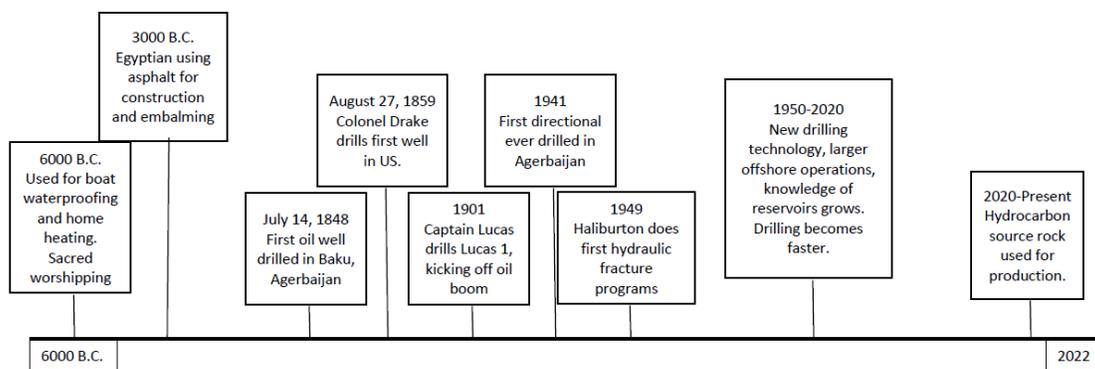


Figure 10: Major technologic advancement of the petroleum industry timeline. Developed from SPE history of industry timeline.

Drilling techniques and equipment rapidly improved allowing for wells to be drilled faster and deeper. This new equipment paved the way for the first directional well to be drilled in Azerbaijan in 1941, by Soviet driller Alexander Grigoryan. Eight years later, 1949, Haliburton Services conducted the first commercial hydraulic fracture programs in Oklahoma and Texas. In the following 70 years, technology in the industry begins progressing faster. New drill bits and reservoir fracture techniques are developed, offshore production begins, and the scale of the industry is no longer limited to land masses. Larger, more efficient drilling practices emerge allowing for deeper and larger well bores. Formations that were thought to never produce economic quantities of oil and gas are now deemed profitable due to the modern technologic capabilities (SPE, 2022).

The petroleum industry has always been an evolutionary marvel. From ancient civilizations collecting oil from the surface to today's mile long horizontal wells in tight formations, the industry is ever-changing and learning. The generation of oil and gas started as a mystery but now wells are tapping into the source material. The next step in the industry's evolution might be speeding up the conversion process of source material to a producible state, or synthetically generating hydrocarbons.

2.2. Kerogen Conversion

A study is being conducted on the formation of hydrocarbons based on the decomposition of kerogen via pyrolysis and low and high temperature oxidations in Russia's largest shale formation, the Bazhenov formation. A simple retort analysis is being conducted on multiple powdered core samples from Bazhenov locations (Bondarenko et al., 2017). A retort analysis is the process of heating a sample and collecting the hydrocarbon condensate that is released. This is commonly done in a heating element (oven) with a glass collection device for the released

fluid. This method is used on core samples to determine average fluid volumes contained within the core. Other testing methods involved in the study are simultaneous thermal analysis (STA) and thermomicroscopy (Bondarenko et al., 2017). Simultaneous thermal analysis is the application of both scanning calorimetry and thermogravimetry to a sample. In this process, the change in mass and changes due to temperature are being observed. Thermomicroscopy involves the use of a Carl Zeiss Axio scope A1 microscope with a heating stage to monitor the macrostructure changes of a sample. Results of the study have shown that there is a maximum temperature range that hydrocarbons can be generated and that more mature kerogen has a lower ability to generate hydrocarbons than that of a younger kerogen (Bondarenko et al., 2017).

As a result of this study, younger kerogen seems to be the desirable choice for performing an in-situ conversion process (Bondarenko, 2018). Attempting a kerogen conversion on a source rock with a small hydrocarbon yield would be inefficient to convert and not economically sustainable. For kerogen conversion, younger formations with a higher OM content should be used.

It is possible to evaluate the volume of recoverable oil from the oil shale. The evaluation requires properties of the formation, OM content, and grade, for example (Allix et al., 2010). These properties are not constant making the evaluation difficult and often inaccurate to a certain degree. Allix et al, 2010, did a surface retort analysis of a 100-g Green River formation sample then conducted a fluid analysis of the generated fluid. The sample was crushed and sieved through a 2.38-mm mesh screen. This crushed material was then heated to 500°C and held for 40-min. The released vapors were collected and distilled in a centrifuge. These separated values were used to determine the percentages of oil, water, shale residue produced, and specific gravity

of the oil. They determined an average oil yield from kerogen of 100 L/megagram (Mg) (24 gal/ton) (Allix et al., 2010).

Using the average yield value, 100 L/Mg, and the average 11% OM by weight content of the Bakken shale, the expected fluid yield can be estimated. The values in Equation 2 are scaled down to represent the size of core samples used in this study.

$$M_{sample} * \%_{OM\ content} = M_{OM\ in\ sample} \quad 1$$

$$M_{OM\ in\ sample}(g) * Yield \left(\frac{mL}{g} \right) = V_{oil\ from\ sample}(mL) \quad 2$$

Although simple, these equations will be useful in predicting the volumes of oil able to be produced from the core samples in this study. This volume calculation does not account for gas loss but as the study and equipment is in the preliminary stages, that should be acceptable.

2.3. Current In-situ Studies

Converting kerogen into producible hydrocarbons is difficult because there are currently only two methods of bringing the material and the conversion tools together: (1) The formation would be mined, brought to the surface, and sent to another site for conversion into hydrocarbons; and, (2) The more desirable method is to convert the kerogen in place and extract the generated hydrocarbons (Bondarenko, 2018). The In-situ method is more desirable because it would be more economic and less impact environmentally. In many locations, the kerogen bearing formations are too deep to mine. Some In-situ studies include:

1. Thermogas by RITEK Oil Company, Russia;
2. ElectroFrac by ExxonMobil Corporation;
3. In Situ Conversion Process (ICP) by Shell Oil Company;
4. Conduction, Convection, and Reflux process by American Shale Oil; and,
5. CRUSH in situ process by Chevron.

Excluding the Russian Thermogas project, these studies are being tested in the Green River Formation in Wyoming. The formation has little recoverable oil but does have a high estimate of potential oil. The outcome of these projects is to release hydrocarbons from the existing kerogen rich shales through different methods of heat injection.

2.3.1. The In-Situ Process

In-situ is a Latin phrase that means “in the natural or original position or place” (Merriam-Webster, n.d.). In terms of kerogen conversion, the OM must be left in place which would eliminate the previously stated mining method. One report (Johnson et al., 2004) discusses that an In-situ conversion process would be economically sustainable if the formation had a permeability large enough for a conversion process to flow through the rock, convert the kerogen, and push generated fluid out, shown in Figure 11.

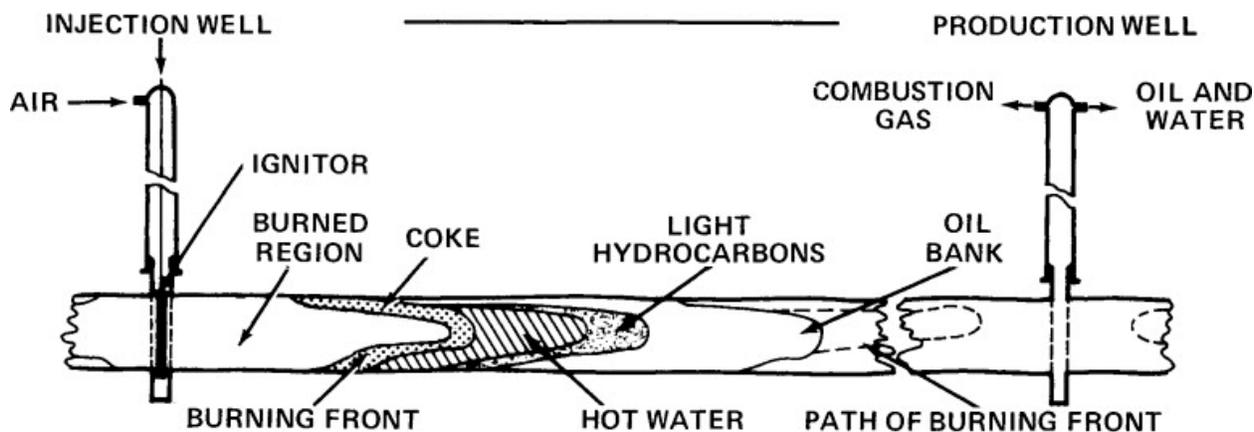


Figure 11: In-situ combustion process (Speight, 2013).

A traditional in-situ method would be to hydraulically fracture the rock, inject air, then begin a combustion reaction that would move through the formation. The report mentions that in this process the combustion front is difficult to control, and the filtration of the generated fluid is not monitored. This added difficulty would leave part of the formation unheated and thus

unaffected by the process. Additionally, due to the combustion, some material is burned and left as coke instead of being converted and extracted.

2.3.2. Thermogas Project by RITEK Oil

The Thermogas process integrates thermal and gas enhanced oil and gas recovery techniques to increase production and attempt kerogen conversion through high temperature air and water injection. RITEK Oil is conducting this study in the Sredne-Nazymkoye oil field in Russia. This project has shown that the hydrocarbons gas, CO₂, and nitrogen content increase significantly as the project continues. An upside to the project was that the oil production drastically increased (Bondarenko, 2018). This increase in production was due to the large increase in reservoir pressure. With these additional gases in the reservoir, the operators started seeing evidence of more oxidation reactions on recovered cores. These reactions were observed by the change in appearance: more red coloring, absence of smell, and trace amounts of hydrocarbons (Bondarenko, 2018).

2.3.3. Exxon's ElectroFrac Project

The ElectroFrac project uses conductive proppant to fracture the formation. When a source of electricity is introduced to the proppant, the formation is also exposed to that source of electricity, as shown in Figure 12. By adding energy to the system, the formation is heated which invokes the pyrolysis process. Using this method, a new kind of proppant must be used and a method of getting an electrical current to the proppant must be added to the well completion procedure (INTEK, 2011).

The continuous addition of heat to the formation is an interesting concept because there is a limit to the oil generation temperature. A thermostat might be used to control the electrical current introduced to the formation, therefore controlling the temperature range, otherwise, the

additional heat would start to combust the formation which would cause the destruction of hydrocarbons and be undesirable.

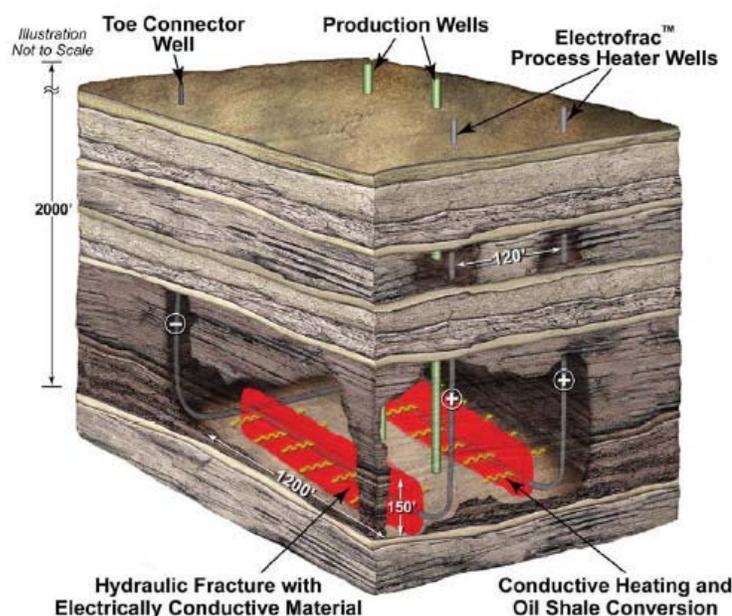


Figure 12: Visual explanation of Exxon's ElectroFrac project showing the conductive proppant used in fractures to heat the formation (INTEK, 2011).

2.3.4. ICP Project by Shell Oil

The In-situ Conversion Process (ICP) is a project that was conducted by the Shell Oil Company in the Piceance Basin, in Rio Blanco County, Colorado. In this method, electric heaters are installed beneath the formation to slowly increase the formation temperature, shown in Figure 13. The current development shows that this technique produces about 1/3 gas and 2/3 light oil which reduces the number of stages in the location's processing facility (INTEK, 2011).

This project has the same concerns that the ElectroFrac project had; the system must be controlled by a formation thermostat. If not, the formation might get too hot for the kerogen to convert resulting in some burned rock and no oil.



Figure 13: Shell's In-situ Conversion Process project visual representation (INTEK, 2011).

2.3.5. AMSO CCR Project

Conduction, convection, and reflux is a process being tested by the American Shale Oil company (AMSO), shown in Figure 14, in the Piceance Basin, Colorado. AMSO is using advanced drilling techniques to place heaters below a production well. Then using micro-fractures, heat is transferred through the formation via convection. As the kerogen converts into gas and rises in the formation, it then refluxes (flows in the direction opposite to normal) and sinks back to the production well. AMSO then uses the produced gases as a fuel for the downhole heater (INTEK, 2011).

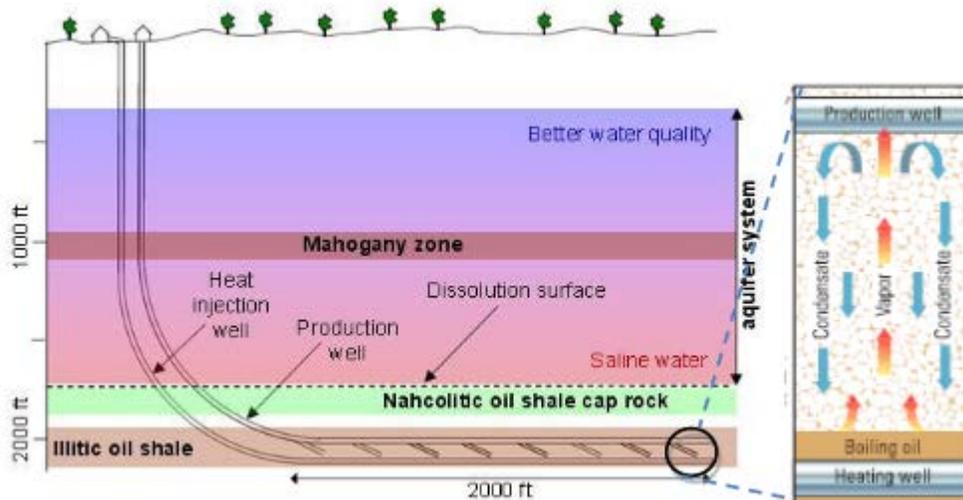


Figure 14: AMSO CCR project with flow diagram (Allix et al., 2010).

Conceptually this project is appealing because it will be generating its own fuel for further heating. Regardless of the conversion process, the process requires additional drilling and may need additional hydraulic fracturing. These additional services add to the project total cost, extending the payout period. The process should show an increase in overall production due to the fluid property changes with a temperature increase.

2.3.6. CRUSH Project by Chevron

Chevron's CRUSH (Chevron recovery and upgrading oil from shale) in-situ project is using high pressure carbon dioxide to crush the formation to rubble. Then using downhole heaters, the kerogen rubble is heated in an attempt to convert the kerogen to hydrocarbons, any hydrocarbon gas produced is then converted to fuel gas to continue the process of generating hydrocarbon liquids. Figure 15 diagrams the high-pressure injection method Chevron is studying.

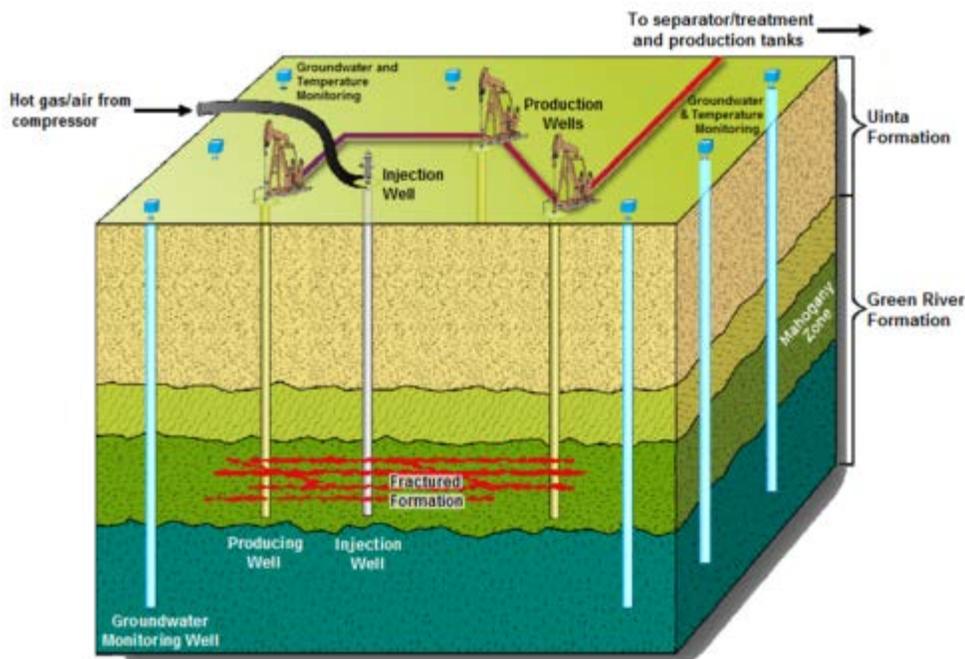


Figure 15: Chevron's CRUSH project of In-situ retorting (INTEK, 2011).

Regarding formation integrity, if parts of the formation are being turned to rubble is there a collapse possibility and would the collapse of the formation compact the rubble to a point where the difference between formation and rubble is indistinguishable. If the formation rubble is compressed further does the permeability decrease and what is the effect of the additional heat? These questions could make the project non-successful and reduce the production capability of the well.

2.4. Ultrasonic Waves for Enhanced Oil Recovery

Preceding the thought of converting kerogen, a study was done to see if ultrasonic waves could be used as an enhanced oil recovery (EOR) technique. Abdulfatah (2018) discusses the process and results of using ultrasonic waves to improve oil production in low permeability reservoirs. The ultrasonic wave should help overcome the interfacial tension between water and oil, aid the mobilization of oil, and reduce capillary pressure (Abdulfatah, 2018). Along with

laboratory experiments, numerical simulations were used to predict water flooding performance and the effectiveness of ultrasonic waves in a porous media. The study concluded that the use of ultrasonic waves in the reservoir could increase recovery up to 50% and significantly reduce the residual oil saturation with the use of a water flood (Abdulfatah, 2018). Although the study of ultrasonic waves was conducted in a conventional reservoir, >20 millidarcies (md) permeability and >15% porosity, the concept is akin this study. A key difference is that instead of trying to increase production by pushing the oil out, this study is attempting to generate more hydrocarbons by converting kerogen using microwaves.

3. Equipment and Methodology

3.1. Samples

Six core samples from the Bakken formation were used in this study, Figure 16. Three of the samples were from the Upper Bakken, and three samples from the Lower Bakken. Cores were collected from the Muller No. 1-21-16H well near Williston, North Dakota. The core samples were provided to this study from the North Dakota Geological Survey. Each core measured about 4.3-cm in diameter and 4.6-cm in height, with an average mass of 120-g. The cores were naturally fractured into multiple pieces allowing for testing of smaller fragments.



Figure 16: Bakken core sample jars with labeled depths.

A thin section description of core sample 9660.5 from the Lower Bakken shale details the lithology as organic-rich, silty shale with a grain density of 2.31-g/cc. The cores have a clay-rich matrix with an average grain size of 0.02-mm. It is important to note that there is an abundance

of pyrite found evenly distributed across the core. The thin section description is included in Appendix A: Thin Slice Description.

3.2. Establishing A Control

To determine if kerogen conversion was possible using EMR, a control needed to be performed. This control was designed to establish if the conversion is caused by heat or by exposure to microwaves. A core sample was placed in an oven and cooked at 150°C for 10-hours. Any significant change in mass (>5%) and visible fluid drainage would show that hydrocarbon generation was occurring. These results are compared to the microwave results.

The oven used was a Quality Lab Inc. Model 30GC Lab Oven, shown in Figure 17. At the top of the oven is a thermometer to verify oven temperature. The core was placed on the center rack for the test to ensure it was equidistance from any heat sources and the walls.



Figure 17: Quality Lab Inc. Model 30GC Lab Oven with door open.

3.3. Microwave Apparatus

The microwave portion of this research began with a basic kitchen microwave appliance, shown in Figure 18. This microwave was made by Whirlpool and has a power output of 1100 Watts, emitting microwaves with a wavelength of 12.23 cm and a frequency of 2.45 GHz. Additional specifications are included in Appendix B: Microwave Data. The power for this device is a standard 120V AC power supply.



Figure 18: Kitchen microwave appliance used in core testing for synthetic generation of hydrocarbons study.

Mentioned in Section 1.4, Microwaves, this appliance has a metal grid over the glass window on the door. This made observing the sample difficult during testing. Some observable moments were flares and sparks when the samples would emit them. This appliance also had a rotating tray in the cooking chamber. The rotation of the object in the appliance is crucial to prevent hot spots. As the microwaves are released from the waveguide, they reflect around the cooking chamber. The waves tend to reflect in patterns, impacting the sample in common points. When the waves bombard a single spot repeatedly, a hot spot is generated, leaving other sections cooler. To prevent this, the sample is rotated to expose more of the surface to these common reflection points, shown in Figure 19. Through object rotation, the sample is more likely to heat

evenly increasing the potential for success. The rotation of the core does not accurately represent an in-situ environment, but will be used until a better designed testing apparatus is available.

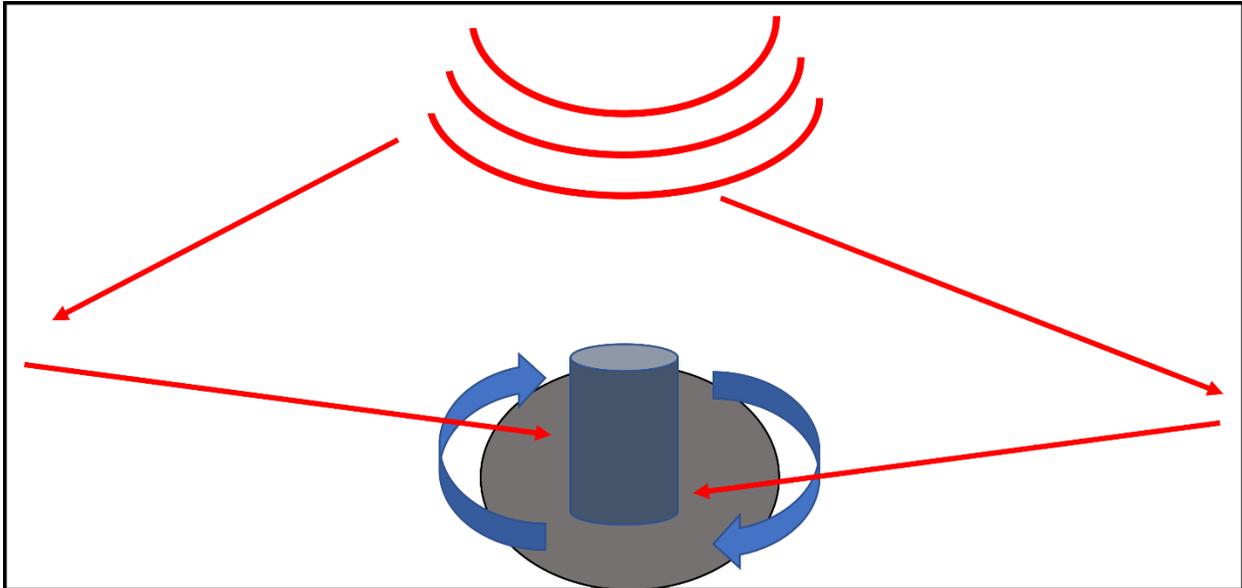


Figure 19: Microwave emittance and reflectance impacting sample. Demonstrating object rotation to prevent hot spots.

3.4. Methods of Measurement

Before testing, the cores were measured to establish an approximate volume for use in energy absorption calculations. Prior to individual tests, the samples were massed to establish a starting point. The mass was then monitored throughout experiments to monitor mass loss from conversion and fluid drainage. At the start of each test, the core's surface temperature was measured with the laser thermometer shown in Figure 20. This thermometer was chosen because of its quick read and update time. The current temperature would update in 1-second intervals and the max temperature detected was also noted on the screen.

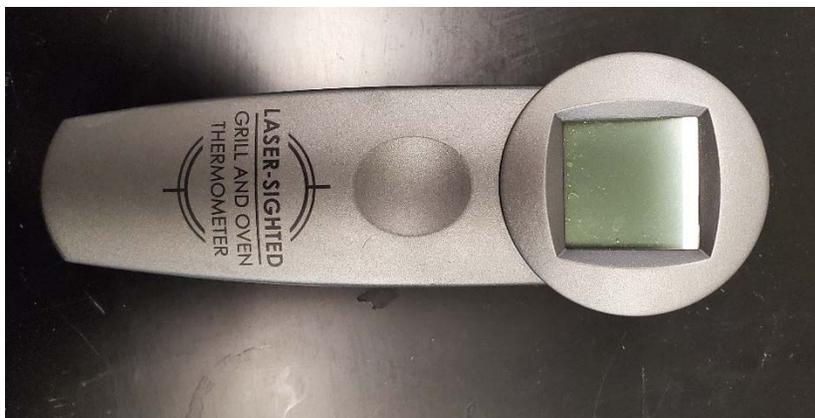


Figure 20: Laser thermometer for measuring surface temperatures of solids.

One goal of the study was to extract fluid from the core sample for a fluid analysis. To perform a fluid analysis, the extracted fluid would need to be collected uncontaminated. For the first test, the fluid volume was thought to be small, thus, the collection device had to be small so that the volume measurement would be accurate. This posed a challenge as a glass beaker was too large and the graduated cylinders available were too small to hold the core. Aluminum Petri dishes were available but would cause sparking issues when placed in the microwave appliance. An idea proposed was to use a paper towel. Common industrial paper towels are porous materials that are designed to collect liquids and hold them. They are flexible and can conform to any shape. A paper towel would work well because the start and finish masses could be recorded. Evidence of fluid collection would then be a wet feeling paper towel and the mass had increased.

Testing the core samples in water became part of the testing process to evaluate if total submergence in a different fluid had any impact. With this process it became necessary to monitor the water temperature. The laser thermometer was not capable of providing accurate measurements of a liquid. To measure the temperature of the water, a probe thermometer was used, Figure 21. This thermometer was slightly slower than the laser thermometer, about 1.5-second response time, but it was able to provide results before the water bath cooled.



Figure 21: Probe thermometer, used to measure liquid temperatures.

In the water bath tests, 100 mL glass beakers were used. This volume of water was small enough to be heated quickly yet still fully submerge a core sample. They did boil over easily which made evaluation of how much fluid the core absorbed difficult. Fluid remaining in the beaker was measured post-test regardless of spillage.

For all tests, the following criteria were set as values of significant change:

- Mass change: difference of 0.10 g or 0.2%;
- Temperature change: final temperature within 80 and 110 °C; and,
- Fluid development: ability to see and touch surface fluids, fluid/gas bubbles present when examined under microscope.

3.5. Proposed Procedure

The first experiments were conducted to develop an understanding of how the materials would react in the microwave appliance. The core was photographed, massed, and the surface temperature measured. The starting core temperature was room temperature, near 22°C. The core was placed in the appliance and cooked. When the cook time was up, the core surface temperature was immediately measured with the laser thermometer. This measurement was taken as soon as possible to ensure minimal temperature loss for future analysis purposes. After the

temperature was recorded, the core was massed. The core masses were used to verify gas development and non-visible fluid drainage.

Post massing, the core was photographed both overall and with a microscope to examine micro-development spots. Microscope magnification values of 2x, 4x, and 6.3x were used for core surface evaluation. Finally, the paper towel was massed and examined for presence of fluid.

3.6. Areas of Focus

To determine if hydrocarbons were being generated, three areas of focus emerged:

1. Did the mass change significantly (>0.1 g or 0.2%)?
2. What was the change of existing fractures, did new fractures open, and did any fractures cause the core to split?
3. What were the properties of any fluid that developed either on the surface of the core or collected separately?

With theoretical fluid development being low, examining the core for fluid was decided to not be the primary method of determining success. This led to using the mass as a deciding factor for fluid development. Due to kerogen having a higher density than oil and gas, fluid development will be able to be assessed through the loss of mass. Mass loss could also occur if the growth of a fracture causes the core to split. To eliminate mass error caused by the loss of small core fragments, the sample was contained in a Petri dish.

When most substances experience a phase change, they expand when temperature increases, and shrink when temperature decreases. The same process is predicted to occur during this conversion of kerogen. Due to this process, the newly generated fluids would need somewhere to go. If the fluids cannot escape through the pore space it will make its own space, by expanding the rock, leading to fractures. Fractures can occur because there is minimal

pressure on the exterior of the core in the testing apparatus. Most fractures would be on the microscale, but some might grow large enough to view with the naked eye. Some fractures may grow to a point where they would cause the core to split into multiple pieces.

With the goal of the project to develop a method of synthetic generation of producible hydrocarbons, it is critical to examine the properties of the developed fluid. The ability to produce a liquid is dependent on whether the fluid can separate from the formation and flow through the reservoir.

4. Results

Five experiments were conducted to develop an understanding if EMR can convert kerogen to hydrocarbons in short term exposures. First, a control was established to understand what happens to the shale cores when exposed to heat. The second series was exposing a core to microwaves when the appliance was set to 40% power. After that series, a core was then tested with the microwaves at 100% power. Fourth, a core was tested in a water bath with the microwave at 50%. Finally, a core was tested in a water bath with the appliance set to 100% power output.

4.1. Analysis of control

Since thermal maturation temperatures need to be between 100 and 150°C for oil development, it seemed practical to heat the cores for a period within this temperature range. The oven was preheated to 150°C. A Bakken core was measured for mass and temperature, then placed in an aluminum petri dish to collect draining fluids. The core was left in the oven for 10-hours and checked at 1-hour intervals. Data recorded from the control test is listed in Table 1.

During the 10-hour cook time, the core showed a continuous loss of mass. This would be a good sign of kerogen conversion due to hydrocarbon generation, but there were no signs of any fluid generation. This analysis led to the hypothesis that water vapor had been absorbed from the air and trapped in the surface pores and clay particles of the core. To test this theory of water loss, the core was left in the open for 2-days to see if it would adsorb water vapor from the atmosphere. After the wait period, the core was massed to determine if any adsorption had occurred. The core had almost returned to its original mass. Leading to the conclusion that some water vapor existing on the core was lost during experimentation.

Table 1: Control data and core description

Core Number:	9602.5	
Core Description:	Selected largest piece of heavily fractured core. Felt dry. Slight oily smell. No significant fractures	
Interval	Mass	Temp
[hrs]	[g]	[°C]
0	42.115	20.9
1	42.021	137.4
2	41.961	138.6
3	41.928	139.9
4	41.912	135.4
5	41.895	131.8
6	41.882	142.4
7	41.879	142.6
8	41.869	139.0
9	41.869	141.2
10	41.860	142.7
2 days	41.920	20.9

Although the oven was set to 150°C, it was operating at a range of temperatures with a low of 131°C and a high of 143°C. This control set some low expectations of kerogen conversion for the rest of the study. Fractures did not grow or form during the heating process. In each examination there was no crude smell or evidence of liquid developing on the core's surface. It is possible that hydrocarbon gases did develop and dissipate between examinations. The formation of gas is plausible as the mass did fully recover after the wait period. If gas did develop there should have been some associated liquids as there was in the dry core tests.

4.2. 40% Power Analysis

The first series of tests were with the microwave at 40% power. Running the appliance at this power rating does not actually output 40% power, it outputs 100% power, 40% of the time it is being run. In this style, the magnetron operates for a few seconds then turns off for a few seconds, then repeats the cycle until the time is complete.

For this series of experiments, half of Upper Bakken core 9600.3 was used. Starting the experiments, the core was dry, minimal crude smell, and had minimal fractures, shown in as Figure 22. Upon closer examination, there were reflective speckles across the surface. An area in the upper left region of the core, resembling a fingerprint like ripple, had the most reflective surface on the core. This was thought to be a potential hot spot during testing.



Figure 22: Bakken core, 9600.3 #2 prior to EMR exposure.

This exposure series was comprised of 3 tests that showed minimal fluid generation and fracture growth. After each test, the core measurements were made, and the core was thoroughly examined for fluid development. In Table 2, the results show that the core can be rapidly heated, but with minimal mass loss. The 90-second experiment heated the core above the oil generation temperature range. In an attempt to stay within the desired hydrocarbon generation temperature range, the cook time for Experiment-3 was reduced to 75-seconds. This resulted in a more desirable end temperature, but also showed the lowest mass loss of all the experiments.

Table 2: 40% power Bakken core test data

Test #	Time	Start Temp	Start Mass	End Temp	End Mass	Δ Temp	Δ Mass
	(s)	(°C)	(g)	(°C)	(g)	(°C)	(g)
1	60	21.2	53.159	74.3	53.102	53.1	0.057
2	90	24.6	53.112	206.6	53.045	182	0.067
3	75	24.4	53.061	108.2	53.012	83.8	0.049

During Experiment 1, a flare developed slowly, then was ejected from the core. Upon examination, this flare was generated within a new fracture. The hotspot caused a spur to grow out of the fracture, shown in Figure 23. The spur was approximately 2-mm tall and 4-mm wide. It had a metallic appearance and appeared to be very porous when examined through a microscope. Above the spur appeared to be a fluid development spot. Most of the area was determined to be burn residue from the flare, but two small areas showed gaseous bubbles.

**Figure 23: Bakken core 9600.3 #2 spur analysis.**

The observation of bubbles led to an attempt to see within the fractures without splitting the core. Using a 6.3x magnification, small bubbles on the interior of the fracture were observed. To save the core for future experimentation, no analysis of the bubbles was attempted.

Subsequent testing with the same core, Experiments 2 & 3, showed similar results: minimal mass loss, significant fracture growth, and no collectable fluids on core surface. The small surface fluid development spots noted in Experiment 1 had grown but were still too small for a sample to be collected. After the three experiments, it was determined necessary to split open the largest fracture to examine the hidden fluid development within. The fracture interior was examined under a microscope and more gaseous bubbles were found, shown in Figure 24. The gas was entrapped in an iridescent viscous liquid. The bubbles were moldable and would slightly deflate after cooling. These bubbles are the assumed cause of the fracture growth seen on the core during testing.

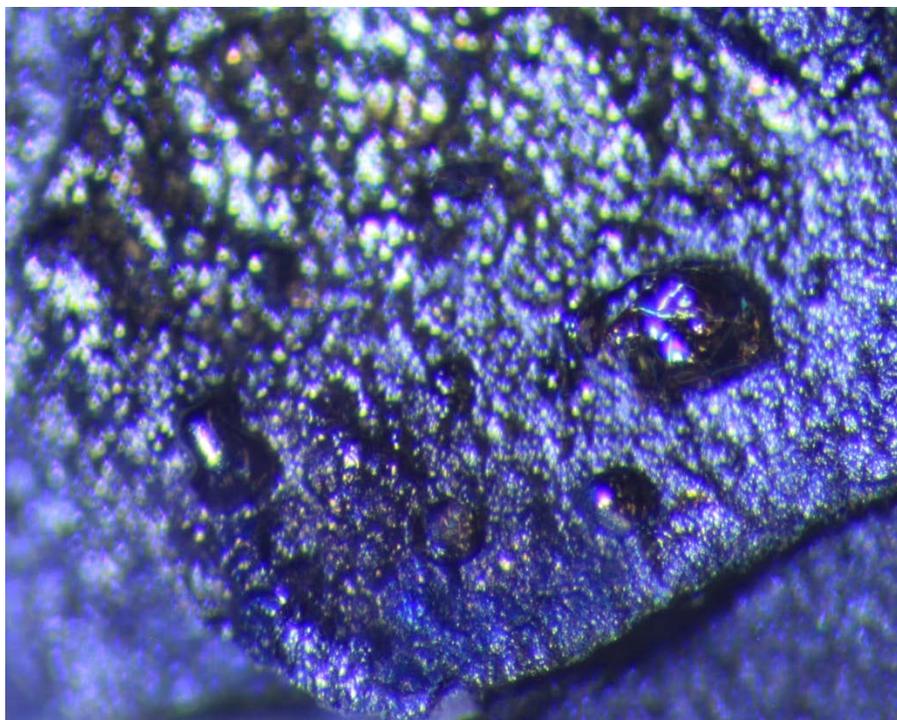


Figure 24: Gas bubbles found in fracture interior of core 9600.3 #2.

Final notes from the 40% power series were that 90-seconds was too long of an exposure time. The core was exceeding the desired temperatures and resulted in the smallest amount of change compared to the shorter exposure time trials. During Experiment 2, the fracture space did not change and there was no change in the volume of surface drainage. These results are curious because to achieve this temperature, the core must pass through the desired temperature range. In passing through this range, some fluid should have developed. Results from the test lead to the assumption that the core quickly passed through the 100-150°C range causing minimal generation to occur. The most change and fluid development occurred when the core was in the 100°C range. Experiment 3 was able to stay within the temperature range but was not able to generate productive quantities of hydrocarbons.

Although a large volume of liquids was not developed during this experiment series, some gas did develop and was evident by the bubbles and mass loss. Methane was assumed to be the gas developed and released from the Bakken core. The total mass lost, 0.147-g, and the density of methane, 6.68×10^{-4} -g/cm³, at normal conditions, 20°C and 14.7-psi, was used to calculate the volume of gas that developed and escaped the core (Whitston & Brule, 2014). This volume of escaped gas was found to be 220-cm³. Although hydrocarbon liquids did not significantly develop, a lot of gas did.

A challenge with this method of kerogen conversion became apparent from these experiments. A microwave will continuously increase the temperature of an object. For this conversion to occur the core needs to be kept in the thermal maturation range which will be difficult to do in a microwave appliance.

4.3. 100% Power Analysis

After semi-successful results of the 40% power experiment series, the 100% power series looked promising. This series used the other half of core 9600.3. Figure 25 shows the core before EMR exposure. This core sample was initially wrapped in a paper towel for testing. It was to collect any developed fluids draining from the core.



Figure 25: Bakken core 9600.3 #1 prior to EMR exposure.

Some key features of this sample that may be important are the perpendicular fracture on the right side and the striations laterally crossing the core. This sample had less reflective spots than the core sample used in the 40% power series, so metallic reflectance was of less concern. The experiment series had 4 tests that resulted in the data in Table 3. For Experiment 2, the core was given time to return to near room temperature resulting in a lower end temperature. The core was not given time to cool off for Experiments 3 & 4, this resulted in higher end temperatures and lower mass loss.

Table 3: 100% power Bakken core test data with paper towel data

Test #	Time	Start			End			Δ Temp	Δ Mass
		Temp	Mass	Towel	Temp	Mass	Towel		
	(s)	(°C)	(g)	(g)	(°C)	(gm)	(g)	(°C)	(g)
1	27	22.6	69.43	1.71	97.0	69.24	1.78	74.4	0.19
2	30	24.1	68.99	1.78	70.7	68.88	1.77	46.6	0.11
3	30	64.8	68.91	1.76	157	68.82	1.72	92.2	0.09
4	60	87.0	68.82	1.76	168.8	68.14	1.72	81.8	0.04

Test-1 started the series in a concerning way. The test was supposed to be a 30-second cook time. At 27-seconds a loud popping sound emanated from the appliance and the test was stopped for safety concerns. After examining the core, the sound was assumed to be caused by the formation of a gas bubble resulting in the core fracturing and splitting apart. On the surface exposed by the new fracture was a significant fluid development spot, shown in Figure 26. On the bottom edge of the development spot is a large bubble. Surrounding the large bubble is a lot of tiny bubbles that were difficult to identify even on maximum magnification in the microscope.



Figure 26: Bakken core 9600.3 #1 Test-1 fluid development.

These bubbles were made of a similar fluid to that of the 40% power series. A major difference was the volume of fluid. The amount of fluid, although still small, was able to be collected on the end of a cotton swab. Not enough fluid was collected to run a fluid analysis with the available equipment, but from the cotton swab sampling it was easy to see that the fluid was very viscous. A flame was held to the cotton swab to see if the developed fluid was flammable. The fluid did ignite then was quickly engulfed by the flame created from the cotton swab. The same test was performed with the bubbles on the core sample to see if they would create a flash flame. They too flared up and quickly dispersed.

In the first two tests, the cores started at near room temperature. These tests resulted in greater fluid development and fracture growth. For Experiments 3 & 4, the starting core temperature was significantly higher due to the lack of a cooldown period. Experiments without the cooldown period were attempted to examine how the fluid generation and system

temperature would change. This resulted in higher ending temperatures outside of the thermal maturation zone and not generating as much fluid as previous experiments. The core also did not lose as much mass as the lower temperature tests. A notable change was the fracture growth and new fluid spots. Prior to testing there were no large fractures and four microfractures visible under the microscope. Figure 27 shows the increase in the number and size of fractures after Experiment 4. Notice that there are small drip lines towards the right side of the large fracture. Upon closer inspection, enough fluid appears to have developed within the fracture and started to leak out through the high permeable fractures.



Figure 27: Bakken core 9600.3 #1 fracture growth.

After Experiment 2, the core sample fractured into two pieces. This made keeping the sample in one piece for testing difficult but resulted in greater fluid development. Unlike the first fluid development zone, Figure 26, less bubbles were present in the later tests, shown in a wider

scale in Figure 28. There was also a larger flare spot but no spur growth as there was in the 40% power experiment series.



Figure 28: Larger fluid development zones on Bakken con 9600.3 #1.

On the exterior of the core, non-fractured side, more gas bubbles developed. The bubbles were larger in size and started dispersing from the core sample. Due to the high viscosity of the newly developed fluid some of the bubbles remained after cooling, shown in Figure 29. As before, when a flame was held to the bubbles, a flare up occurred.

After consideration of gas development, the evolving gas should be run through a gas chromatograph. Doing so would allow measurements of the change in air composition before and after the test. This measure of change would allow for an analysis in how much gas is being generated and a comparison to the mass loss. Running the evolved gas through a chromatograph was not done because the gas was not of interest at the time. The conversion process was being

tested to determine the liquid oil generation potential. Mass lost during the experiment does not account for the liquids generated since they are not extracted from the core. The change in mass can be associated with the development and release of gas from the sample. At normal conditions (20°C and 14.7-psi), the density of methane is 6.68×10^{-4} -g/cm³ (Whitston & Brule, 2014). Converting the total mass lost to a gas volume, 643.7-cm³ of methane were generated.

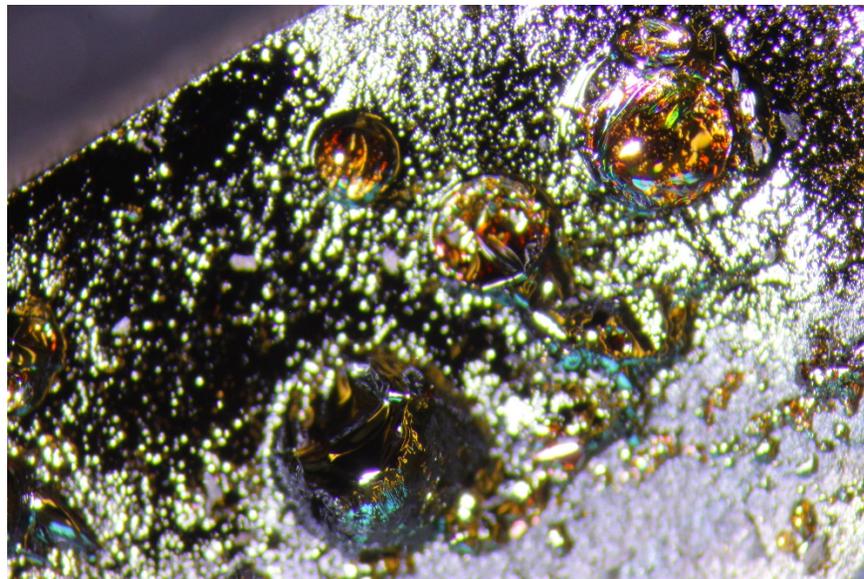


Figure 29: Large gas bubbles developed in Bakken core 9600.3 #1.

The paper towel did cause some issues for this series of experiments. It did not have an apparent impact on the fluid development in the core. The main issue with the paper towel was caused by the hot spots on the core. Hot spots on the core caused parts of the paper towel to burn, creating large amounts of smoke and increased the fire hazard during testing. On multiple instances the paper towel would still have embers on it when the sample was removed from the microwave appliance. For this reason, the paper towel was removed from the procedure for the final test. After testing without the paper towel, there was no obvious difference in the fluid development with or without the towel.

4.4. 50% Power in Water Bath

After positive results from the 40% and 100% power dry core tests, it was necessary to determine if the same results could be achieved with the sample in a water bath. In theory, any developed fluid would transfer from the surface of the core to the surrounding water. Then, by difference of densities, the presumed oil would separate from the water and be measurable. These observations and measurements were not done because no oil was present in the remaining water within the beaker. The water bath tests were first conducted at 50% to see how low power would compare to the later high-power testing. A slab from the already fractured Lower Bakken core sample 9654.5 was used for this series of tests, shown in Figure 30. Like the other samples used, the core was dry, minimal crude smell, and had no visible fractures.



Figure 30: Bakken core sample 9654.5 prior to testing.

With the thermal maturation temperature range starting at the boiling point of water, the possibility of hydrocarbon generation was minimal. Water would boil and splash out of the beaker before raising the temperature of the core to the temperature range the beaker size was determined by the amount of water needed to fully submerge and contain an upright core sample.

A 100-mL beaker was used, this did not leave much space to contain the water, demonstrated in Figure 31.



Figure 31: Core sample 9654.5 #1 in 100 mL beaker.

A larger beaker was not used because the core temperature would be controlled by the heat exchange of water and the core instead of by the microwaves. Simply, the core would be boiled instead of being heated strictly by EMR due to more water being needed to fully submerge the core. This was not within the scope of the project, eliminating this concept.

As the water began to boil around 90°C, it would bubble out of the beaker spilling onto the microwave appliance's rotating tray, leading to more of the core being exposed to the EMR. This boil over situation was not an issue during Experiments 1 & 2 as the water temp did not get high enough to start boiling, shown in Table 4. Another factor to consider is material contact. The temperature of the water is also affected by the temperature of the core sample. The boiling became more aggressive in the later tests, originating around the bottom of the core sample.

Table 4: 50% power Bakken core in water bath test data

Test #	Time	Start				End			
		Core Temp	Core Mass	Water Temp	Water Volume	Core Temp	Core Mass	Water Temp	Water Volume
	(s)	(°C)	(g)	(°C)	(g)	(°C)	(g)	(°C)	(ml)
1	30	22.8	42.193	21.7	65	45	42.214	58.9	65
2	60	20.8	42.198	21.9	65	73	42.224	84.8	64
3	90	21.5	42.209	20.6	65	62.1	42.268	91.9	50
4	90	23.9	42.247	20.9	65	62.7	42.287	90.9	50
5	120	28.7	42.273	28.7	65	64	42.342	93.5	40.5

Once the boiling subsided, smaller bubbles could be seen flowing out of the largest fracture on the core. They did not flow quickly but were also obviously not residual water vapor on the bottom of the beaker. The longer cook times caused longer boiling periods resulting in more spillage. A downside to doing the water bath was the core had to be dried to get an accurate mass measurement. In no tests of this series did the mass ever drop. It was assumed that during the test, water flowed into the core through microfractures. Evidence of the results of this process are shown in Figure 32. This absorbed water was then retained through the massing period. Between tests some water did evaporate resulting in the mass changing between the end of one test and the start of another.



Figure 32: Bakken core 9654.5 #1 water absorbed via fractures.

After completion on this test series, there was no evidence of fluid development. It was unclear as to if any did develop and was washed away in the turbulent water. The residual water in the beaker showed no sign of developed fluids. Water remaining on the surface of the core when removed from the beaker quickly evaporated or was absorbed by the core. Temperatures of the core were lower than those seen when the core was tested dry.

Water that had escaped from the beaker remained on the rotating tray in the microwave appliance. The water was examined and determined to have no developed hydrocarbon fluids present. Showing that at 50% microwave power with the Bakken core in a water bath, not enough energy was entering the system to start the kerogen conversion process.

After the boiling water had cooled there was a slow trickle of small bubbles being emitted from the core. It was unclear as to if the bubbles were trapped air being displaced by water moving into the core or if they were natural gas bubbles. Without a gas capture device, it is difficult to determine. Assuming some volume of trapped gas in the pores exists would be logical and provide some explanation as to why bubbles were forming on the surface during the dry exposure experiments.

4.5. 100% Power in Water Bath

The 100% power water bath test series was expected to have similar results as those seen in the 50% power water bath series. More water would be lost in the longer duration tests and there would be minimal fluid development. For this series, another slab of the 9654.5 Lower Bakken core was used, shown in Figure 33. On the upper-right region of the core there appeared to be a wet spot where fluid could already have been seeping out. After magnified examination, this spot was determined to not be a spot of fluid development. At 6.3x magnification, this material appeared nearly transparent and flakey. There were smaller spots around the core that were similar. The core sample had no visible fractures and smelled faintly of crude prior to testing.



Figure 33: Bakken core 9654.5 #2 prior to 100% power water bath test series.

Through the series, the core never achieved the anticipated thermal maturation temperature. More water was expected to be lost during the process which would have left more of the sample exposed to un-refracted microwaves. As in the 50% power series, a larger beaker was not used in attempt to provide more EMR exposure to the core instead of boiling the core. As the microwaves travel through water they lose energy, meaning less energy is getting to the

core. If less water is in the beaker, then more waves unaffected by the refraction of water can bombard the core. Table 5, below, lists the temperature and mass data for this 5-test series.

Table 5: 100% power Bakken core in water bath test data

Test #	Time	Start				End			
		Core Temp	Core Mass	Water Temp	Water Volume	Core Temp	Core Mass	Water Temp	Water Volume
	(s)	(°C)	(g)	(°C)	(g)	(°C)	(g)	(°C)	(ml)
1	30	21.3	53.687	21.6	65	77.7	53.709	87.8	63.5
2	60	27.5	53.704	20.2	65	77.7	53.756	92.3	35.5
3	60	24.5	53.737	20.2	65	75.3	53.792	92.9	36
4	90	24.5	53.737	20.2	65	75.3	53.792	93.2	28
5	90	27.4	53.762	20.1	65	81.9	53.802	93.6	28

Like the 50% power water bath test series, there were small bubbles being released from the core after the boiling had subsided. In Experiments 3, 4 & 5 the bubbles emerged in larger quantities and flowed at a faster rate than those seen in the 50% series. It is still unclear as to if they were displacing existing gas trapped in pores or newly generated gases. Based on the lack of new fractures on the core, it was assumed the bubbles were formed by absorbed water displacing trapped air, but without a gas capture system in place this is difficult to analyze.

Unlike the dry test series, the mass did not decrease during these tests. Some water was being absorbed by the core during this process through microfractures and surface pores resulting in some clay particle swelling and the core expanding in some regions. After letting the core return to room temperature, some water was released from the core, lowering its residual water saturation and mass. No fracture growth had visibly occurred during this absorption and

loss period. After Experiment 4, surface swelling was obvious. Prior to testing the edge was straight and would rest on the tabletop evenly. After testing, the core would rock back and forth over a point that had swelled on the same edge.

4.6. Soaked Core Testing

Late into this study, an idea of soaking the shale cores in water prior to EMR exposer was proposed and one experiment was conducted. A piece of Upper Bakken core 9602.5 was placed in a water bath and allowed to saturate with water for 1-hour. Prior to entering the water bath, the sample had a mass of 41.924-g. After the soak time, the mass had increased to 42.189-gm, meaning 0.265-mL of water were absorbed by the core's clay grains and empty pore spaces. From room temperature, 18.2°C, the core was then placed in the microwave for 1-min.

After 1-min the core temperature was 157.3°C and the mass was 41.878-gm. Most of the mass loss can be associated with water vaporizing and escaping the core. Though the mass did drop below the starting condition, which leads to the assumption that some gas was developed. Upon examination, the pre-existing fractures had grown significantly most likely due to the water expanding through vaporization, but a portion of the mass loss could also be associated with the gas development. Factoring for the mass of water absorbed then evaporated off, 0.265-g, the remaining mass to be associated with gas development would be 0.046-g. Using the density of methane (6.68×10^{-4} -g/cm³) at normal condition (20°C and 14.7-psi), the volume of mass generated would be 68.8-cm³ (Whitston & Brule, 2014).

Under a microscope at maximum magnification, 6.3x, a very small hydrocarbon development zone was discovered. Only a small drop, about the size of a toothpick tip, of oil was present with no obvious gas bubbles. This spot was discovered when the largest fracture was wedged open to examine the fracture surfaces.

Although this method did not exceed the results shown in the dry core experiment series, it does have potential. The water vapor appears to have kept the core in the oil thermal maturation range for a longer period when compared to the dry core series. This process may allow for longer exposure times without exceeding the desired thermal maturation range. In future studies, this method should be attempted with longer soak times to determine if water saturation has an impact on kerogen conversion and temperature control. The experiment series should also include an experiment series where the cores are saturated in saltwater to better simulate reservoir conditions.

4.7. Data Analysis

Based on the mass of each sample, each core used has a different quantity of potential oil it can convert. It is difficult to quantify the 100% conversion value of kerogen without crushing the sample. In this study, none of the cores were deemed to have converted producible quantities of oil. But the value determining a producible quantity was never defined. If 10% of the kerogen was able to be converted, then the lab value would be quite small. If this process was able to convert 10% of the entire Bakken formation, the quantity would be much more significant. In

Table 6, the OM content in the core was calculated using the average kerogen content of the Bakken formation, 11%, Equation 3.

$$M_{sample} * \%_{OM} = M_{OM \text{ in sample}} = 42.159 \text{ g} * 0.11 = 4.637 \text{ gm of OM} \quad 3$$

Then using the average yield value, 100 L/Mg (0.1 mL/g), the oil yield values were calculated, Equation 4 (Allix et al., 2010).

$$M_{OM \text{ in sample}} * Yield = V_{oil} = 4.637 \text{ g} * 0.1 \frac{mL}{g} = 0.464 \text{ mL of potential of oil} \quad 4$$

Averaging to half a milliliter per core sample, there is not a substantial volume of potential hydrocarbons to convert, but comparatively to the size of the cores any generated volume should be visible.

Table 6: Potential oil yield by core mass.

Core	Mass	OM Content in Core	Oil Yield
	(g)	(gm)	(mL)
9602.5	42.159	4.637	0.464
9600.3 #1	69.43	7.637	0.764
9600.3 #2	53.159	5.847	0.585
9654.5 #1	42.193	4.641	0.464
9654.5 #2	53.687	5.906	0.591

This analysis of oil potential was used to estimate to explain the production potential of a square-kilometer by 5-meter-thick portion of the Upper Bakken. Using a calculated bulk volume of $5 \times 10^6 \text{-m}^3$ and an average formation density of 2.3-g/cc (2300-kg/m^3), the mass of the Upper Bakken section would be $1.15 \times 10^{10}\text{-kg}$ (Hester & Schmoker, 1985). Then, factoring in the average OM content, 11% by weight, the mass of kerogen present is $3.13 \times 10^9\text{-kg}$. Using the average yield value, 100-L/Mg (0.1-L/kg), and the mass of this shale example, $3.13 \times 10^9\text{-kg}$, the 100% yield would be $3.13 \times 10^8\text{-L}$ (1.9-million barrels of oil). Even if the conversion process could only convert 50% of the targeted area, ~1 million barrels of oil is a substantial volume to produce out of 1 square-mile.

Regarding the energy necessary to generate oil from the core sample, the conversion process must require less energy than the microwave outputs. The microwave appliance used in this study has a power output rating of 1100-watts (1100-J/s) with an efficiency of about 80%

(Vollmer, 2004). Accounting for the efficiency, this appliance outputs 880-watts (880-J/s). For the conversion process to be efficient, kerogen needs to absorb less than 880-J/s otherwise energy is being wasted on the formation without any product being generated. In a 30-second exposure, an overall 26,400-J would be introduced to the cooking chamber. The heat capacity of kerogen, an unknown in this project, but using the heat capacity of shale, 770-J/kg °C, an estimate of energy needed can be calculated (Robertson, 1988). Using a core with a mass of 50-g, the energy needed to raise its temperature 1°C would be 38.5-J/°C. To get the core into the desired thermal maturation range, 100°C, the temperature needs to rise by ~80°C. Based on the mass of this core, it would take about 3,000-J to achieve this temperature. This leaves a large quantity of unused energy wasted within the microwave cooking chamber.

Considering the energy output of oil, ~38,400-J equivalent per milliliter, about 0.7-mL of oil would need to be generated from the sample in the 30-second exposure period to produce a neutral energy exchange (University of Washington, 2005). Only one sample in this study, Upper Bakken core 9600.3 #1, has the potential to produce this equivalent energy exchange. If the 100% of the kerogen in this core was converted in the 30-second exposure period, then the energy exchange would result in a net-positive; 26,400-J would be introduced and ~29,000-J equivalent would be available from the newly generated oil. The other Bakken cores would require more energy to fully convert all the kerogen than generated oil could produce.

This method does not actually calculate the energy needed to convert the kerogen, instead it calculates how much energy it would take to heat the sample to the desired temperature. The heat capacity of shale is used instead of the heat capacity of kerogen. Once the energy required to heat the kerogen is calculated, it is then necessary to calculate how much energy is needed to maintain the sample temperature within a few degrees to maintain conversion. A method for

calculating the energy required to continuously convert the in-place kerogen was not found during this study.

5. Discussion

From the use of surface seeps creating long burning fires and the ability to waterproof boats, to deep mile long lateral wells in complex formations, the oil and gas industry has evolved tremendously and is now awaiting the next step. With the rapid expansion of the production of oil and gas across the world, the possibility of generating more is the next step in the industry's evolution. There are few concepts being tested on the synthetic generation of hydrocarbons and fewer in-situ ideas like this process being studied. The ability to use EMR as an energy source for converting young, immature kerogen would be a step in recovering the hydrocarbons buried on this world. This preliminary study sought to answer the study questions outlined below.

- 1) Can kerogen be converted to hydrocarbons using EMR, specifically at the microwave frequency?
- 2) Can the generated hydrocarbons be collected? If so, what collection device will be effective for obtaining and measuring generated hydrocarbon fluids?
- 3) How much energy is required per unit of kerogen for conversion to hydrocarbons?
- 4) How does EMR affect hydrocarbons (existing or generated)?

The following subsections will answer the study questions through an analysis of the data and knowledge gathered from this study.

5.1. Study Question 1

Can kerogen be converted to hydrocarbons using EMR, specifically at the microwave frequency? This study question was not meant to discuss the possibility of kerogen conversion but moreover the curiosity of whether microwaves are powerful enough to perform the conversion.

Through retort analyses, it is known that kerogen conversion is possible if enough heat is added to the system. This study was not only looking at the addition of heat, but the addition of energy.

If refined, this in-situ process could effectively and efficiently convert kerogen to producible quantities of hydrocarbons that can provide more energy than required for the conversion. Dry conversion works, but more research needs to be conducted at this frequency on a larger number of samples to quantify effective depth and rate of conversion. Regarding the range of EMR, a lower frequency, $<10^8$ Hz, might have the ability to penetrate deeper into the formation than a microwave, 10^9 - 10^{11} Hz. Although a lower frequency might not have the power that the higher frequency microwaves have available to convert kerogen to hydrocarbons. Further investigation of frequency on kerogen conversion is needed.

5.2. Study Question 2

If possible, can the generated hydrocarbons be collected? What collection device will be effective for obtaining and measuring generated hydrocarbon fluids? In short, the answer is yes, the generated hydrocarbons can be collected.

The issue with collecting the new hydrocarbons is their ability to flow away from the core sample. So far, in this study, the generated liquids have not gathered in a large enough volume to drip off the core. For a better understanding of fluid collection, more hydrocarbons need to be generated. Through examination of the core post experimentation, gas has no restriction to flowing away from the core. It was assumed that most of the gas generated left the core during the cooking process. In future testing, the gas needs to be collected so that it can be analyzed and quantified.

For gas collection, an airtight container needs to be designed so that gas can be removed during the experiment. A proposed method was to use a bell jar with that has a vacuum pulled on it. Any gas generated would flow out through plastic tubing to the vacuum pump. Then gas would flow through the gas chromatograph. This would allow for real time measuring and monitoring of gas generation.

Collection of the liquid hydrocarbons is challenging because of the small volume of fluid generated. What fluid has been generated has not readily left the sample in successful experiments making a fluid analysis difficult. A proposed idea for analyzing the developed fluid was to use the resources at the Montana Bureau of Mines and Geology (MBMG) at Montana Technological University. The MBMG lab personnel described their process and the difficulties they would encounter when analyzing this developed fluid: (1) not enough fluid had been developed to be processed with their equipment (an Aurora 1030 total organic carbon analyzer), (2) using a carrier fluid is complicated because the chemical makeup of the developed fluid is unknown, and (3) if enough of the developed fluid was available, it could clog up the analysis device. The MBMG lab was not designed for hydrocarbon analysis, they recommended contacting a university department that is more familiar with the organic chemistry of hydrocarbons.

5.3. Study Question 3

How much energy is required per unit of kerogen for conversion to hydrocarbons? In simplest terms, this question is asking what the heat capacity of kerogen is. Through all the literature reviewed, no documentation on the heat capacity of kerogen was found. Some literature was found on the heat capacity of shale. To raise the temperature of a 50-g core to the desired thermal maturation range, ~3,000-J are needed. The microwave was able to exceed this

energy requirement in every experiment. If 0.7-mL of oil can be generated from the core, the energy used would be less than the energy potential which would consider this method efficient. However, only one sample tested had the potential to produce enough oil to negate the energy required for the conversion. Since most of the Bakken core samples could not theoretically generate enough oil, there is an inefficiency in the process. The amount of generative material in the samples cannot be changed, but the efficiency of the microwaves can be improved to reduce wasted energy. With a smaller cooking chamber there is would be more wave bombardment which would increase the efficiency of the heat transfer for the kerogen conversion.

5.4. Study Question 4

How does EMR affect hydrocarbons (existing or generated)? To test the effect of EMR on oil, two comparative 30-second experiments were run with samples of water and Bakken crude. The first analysis used a constant volume of 20-mL. The second analysis was done with a fluid mass of 15.0-g. The change in fluid temperatures is shown in Table 7. The water temperature increased to the near boiling point in the 30-second exposure time, meanwhile the oil temperature was raised slightly.

Table 7: Change in microwaved fluid temperatures.

Fluid Type	Constant	Start Temp (°C)	End Temp (°C)
Water	20-mL	22.9	94.2
	15-g	23.4	93.2
Bakken crude	20-mL	23.6	27.0
	15-g	22.9	26.1

Having a small temperature increase, showed that the EMR will not affect the existing or newly generated hydrocarbons significantly. As more hydrocarbons develop, the temperature

should begin to stabilize around the thermal maturation temperature range, continuing the conversion process instead of stopping due to high temperatures.

6. Future Work

Overall, this study has shown that kerogen conversion using the microwave range of the electromagnetic spectrum is possible. It is still unclear as to why it works and the efficiency of the process, but as a baseline it works. This study has brought up many future routes to experiment with and new aspects of kerogen conversion to investigate. There is a lot of work still to be done to consider the lab portion of the study complete. The following subsections entertain some possibilities for future work.

6.1. Understanding of Heating Kerogen

In the next step of the study, an understanding of the heat capacity of kerogen is required. This study estimated that ~3,000-J are required to heat the core sample to the desired temperature range, but the amount of energy for successful conversion completion is still unknown. Once this value is known, keeping the sample in the target temperature range with EMR will be easier. The output of EMR interacting with the sample will be modified whether the sample needs more heat or less heat.

After developing an understanding of energy needed for the conversion, a comparison of energy out of the magnetron and energy into the core can be used to determine a conversion efficiency. Using this efficiency value, the conversion process can be refined by modifying the energy output to convert more kerogen.

6.2. Designing a Better Testing Apparatus

Naturally kerogen converts to hydrocarbons in an anerobic environment. The current testing apparatus is flooded with air, rich in oxygen. This is a major difference in this study and the possibility of this process working in-situ. To ensure this process will be successful in the formation, it first needs to be tested in an oxygen lacking environment. A proposed idea was to

flood the microwave cooking chamber with nitrogen prior to emitting microwaves. This would work if the appliance door was airtight. Alternatively, the sample could be placed in an airtight glass vessel, filled with nitrogen. Microwaves can easily pass-through glass without losing too much energy. Then the oxygen lacking conversion could occur. However, this method still leads to difficulty measuring the generated gas volume and makeup.

A second solution was to run the experiment in a vacuum. As discussed earlier, the sample would be placed in a bell jar, capped, then had a vacuum pulled on it. This method would require drilling holes in the appliance and running tubing from the bell jar to the vacuum pump, which is easily done. This method would reduce the oxygen in the test chamber and allow the capture of generated gases. The biggest issue with this is being able to take the core sample temperature immediately after testing. This could easily be rectified by fitting a microwave safe thermometer to the core sample.

A third apparatus style that was proposed, was constructing a new device that eliminates the need of the microwave altogether. Either a microwave magnetron or variable EMR emitter would generate waves that would be piped to a fitted sample container. Then the waves could only impact the sample. There would be no reflective space for the waves to bounce off, and no air space for oxygen to remain. Gases and exhaust could then be plumbed through a gas chromatograph for analysis. Liquid hydrocarbons would remain in the sample container for later analysis. This method is the most complex and would require a design and construction period in the next stage of the study.

The experiments done in this study were performed at atmospheric pressure, about 13 psi. Reservoir pressure can exist at and above 3,000-psi. The difference of these two environments may significantly impact the results of this conversion process. A future experiment series should

be designed with the core in a pressurized system. Adding pressure to the core may show that the temperature range of kerogen conversion is significantly higher than that determined in the experiments done for this study. Unlike the second testing apparatus proposed, this fourth apparatus could allow the system to be pressured to near reservoir conditions allowing for a better understanding of how this conversion method would work in the formation.

6.3. Varying Frequencies

The electromagnetic spectrum covers a large range of known frequencies and wave types. The range of microwaves, although large itself, is relatively small compared to the entirety of the spectrum. There are frequencies that have better depth penetration and there some that carry more energy with them. It would be wise to investigate how other frequencies affect the conversion process.

To investigate this, a frequency modulator will be needed in the testing apparatus. Doing this will allow future researchers to experiment with various frequencies for individual experiments or modulate the frequency during an experiment. This could show the conversion happens quicker or more efficiently with one frequency compared to that of microwaves.

7. Conclusion

As hydrocarbon bearing reservoir volumes deplete across the world, the necessity to locate new sources of hydrocarbons rapidly approaches. In response to the need, we evaluated methods of speeding up the natural process of conversion from kerogen to hydrocarbons from known formations such as the Bakken shales. This study was conducted to investigate this synthetic generation process using electromagnetic radiation, specifically microwaves. Current conversion methods require injecting expensive material or extraction of the raw material. These processes increase the operating cost tremendously and have not shown to be economic. The implementation of a microwave emitter into a wellbore completion could be a potential solution.

In this study, the preliminary work for using microwaves to kickstart the conversion process was tested and showed some promising results. In lab scale tests, fluid—whether pre-existing or newly generated—was developed from shale cores using a common microwave appliance. A solid base and direction were developed for future experimentation to continue. This preliminary study aimed to answer the following: (1) can kerogen be converted using EMR, (2) can the lab scale volume of hydrocarbons generated be collected, if so, what collection device fits the needs of this scale, (3) how much energy is required, and (4) what is the effect of EMR on generated or existing hydrocarbons. The conclusions from these study questions are as follows:

- 1) Synthetic generation of hydrocarbons, kerogen conversion, is possible using electromagnetic radiation. It is unclear as to if the range of microwaves is the most efficient range for conversion or not. Leading to a future study question of what is the most efficient frequency, and does it depend on the formation?

- 2) Lab scale developed fluids can indeed be collected. At this point in the project not enough information was known about what was needed to collect this small volume of fluids. Testing of collection devices needs to be done to see which device will best suit the needs of the volume generated. Gas takes up more space and will require some sort of a pressure containment unit. The oil volume is quite small requiring either generating more fluid or a method of cleaning the fluid from the sample.
- 3) The amount of energy required to convert a unit of pure kerogen is still unknown. This study estimated that about 3,000-J are required to bring a room temperature core to thermal maturation temperature. It was recommended to study the amount of energy required to maintain the temperature of the core and continue the conversion process.
- 4) Electromagnetic radiation should have no effect on hydrocarbons based on the polarity of the molecule. Oil is mostly non-polar and therefore should not be greatly affected by microwaves. Regarding the impurities generated through thermal maturation, the effect is still unclear.

To conclude, this study takes a major step forward into using EMR as a tool for the synthetic generation of hydrocarbons. If the process can be refined and made more efficient, it could be a major tool used by hydrocarbon production companies worldwide. Electromagnetic radiation could extend the production lifespan of known source rocks. Even if the kerogen conversion process proves to be ineffective, this study has shown that microwaves are effective at fracturing rock, creating permeable highways for fluid flow to increase production volumes.

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9. Appendix A: Thin Slice Description

G3 Operating LLC
 Muller No. 1-21-16H
 Williams County, North Dakota
 Conventional Core

WFT Labs File: CO-51887

Thin Section Description – General

Sample Depth: 9660.50 Feet
 Sample Number: 1-80

Plate 6

Klink. Perm. (2600 psi): N/A
 Porosity (amb.): 8.9%
 Grain Density: 2.31 gm/cc

Lithology: Organic-rich, silty shale

Texture: Laminated with rare possible small subhorizontal burrows; common compacted spores (*Tasminites*) aligned along bedding (orange; B-C12,F2, photo B); locally extensive pyrite replacement along laminations

Grains: Abundant subangular to angular silt-size (up to 0.05 mm) siliciclastic grains; estimated average grain size = 0.02 mm; monocrystalline quartz (C-D3.5, photo B) > feldspar (potassium feldspar ~ plagioclase) > muscovite > polycrystalline quartz > chert > biotite/altered biotite, heavy minerals (titanium oxide); common, fairly evenly distributed, compacted spores (*Tasminites*; orange; E4,J-K3, photo A; A10,F2, photo B); sparsely scattered compacted agglutinated foraminifera (H6, photo A); trace phosphatic fragments

Matrix: Abundant detrital clay-rich matrix supports grains; appears dominantly composed of detrital clay with interspersed organic matter; clays appear well aligned, with most crystals displaying "unit extinction" under crossed nicols; abundant intermixed small crystals of replacement pyrite and sparsely scattered crystals of replacement dolomite (G2, photo B)

Cements and Replacement Minerals: Abundant pyrite (black; F-G11.5,H6.5, photo B) is fairly evenly distributed as a replacement of matrix, organic matter, and agglutinated foraminifera, and is concentrated along a few laminations; minor authigenic quartz occurs as cement within some less compacted spores; sparsely scattered fine crystalline dolomite occurs as a replacement of matrix; a few relatively large crystals of authigenic apatite occurs as cement in spores (B-C12.5, photo B) or as non-selective replacement (A-B12, photo B)

Pore System: No visible pores using standard petrographic techniques

Magnification: A: 50X B: 200X

10. Appendix B: Microwave Data

