NJIT New Jersey's Science & Technology University

Green Chemistry in Teaching Laboratory: Microwave Induced Reactions

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This Project was undertaken in connection with a settlement of an enforcement action taken by United States Environmental Protection Agency against New Jersey Institute of Technology
Green Chemistry in Teaching Laboratory: Microwave Induced Reactions

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Safety tips:

According to Food and Drug Administration, the maximum amount of microwave radiation that can leak out from the microwave oven can be 5 mill watts throughout its lifetime. It's always considered safe to work at a distance from the oven as microwave energy decreases with increase in distance from its source of radiation. The microwave oven must have two independent interlock systems such that the production of microwave radiation ceases when the door opens. Care should be taken while working with microwave ovens, as microwave radiation can cause serious injuries to human body. Exposure to large amount of microwave radiations can lead to injuries like- burns, cataract, temporary sterility.

- Read all instructions before using the microwave.
- Avoid possible exposure to excessive microwave energy.
- Do not attempt to operate this oven with the door open since open-door operation can result in harmful exposure to microwave energy. It is important not to tamper with the safety interlocks r drill holes with the external body.
- Do not operate the oven if it is damaged. It is particularly important that the oven door closes properly and that there is no damage to the: (1) door (bent), (2) hinges and latches (broken or loosened), (3) door seals and sealing surfaces.
- Take special care to ensure that no damage occurs to the part of the oven making contact with the door or door seals .Do not by pass the door interlocks.
- The appliance must be grounded properly. Ensure that the microwave is unplugged or disconnected from electrical power before reaching into any accessible openings or attempting any repairs.
- Do not test a microwave power generating component without an appropriate load connected to its output. The power generated must never be allowed to radiate freely into occupied areas.
- Ensure that the adjustment of applied voltages, replacement of the microwave power generating component, dismantling of the oven components, and refitting of waveguides are undertaken **only** by persons who have been specially trained for such tasks. The services of a qualified repairman should be sought when any malfunction is suspected.
- Never operate an empty microwave. If you want to practice using it, place a cup of water inside to absorb the microwave energy.
- Keep the inside of the microwave clean. Wipe up chemical spills as they occur.
- Metallic vessels are not recommended for microwave use. Do not use aluminum foil or metallic components.

- Sealed containers tend may explode. Such containers must not be heated in a kitchen microwave with a special pressure sensor are recommended. .
- Do not cover or block any openings on the appliance.
- Do not store a microwave outdoors. Do not use it near water– for example, near a laboratory kitchen sink,
- If materials inside the oven ignite, keep oven door closed, turn oven off, and disconnect the power cord, or shut off power at the fuse or circuit breaker panel.
- Do not use the cavity for storage purposes. Do not leave paper products, cooking utensils, or food in the cavity when not in use.
- Experiments that required the use of volatile solvents and hazardous chemicals are to be used carefully. Fire hazard is also an important consideration when microwaves are concerned.

S. No:	Unsafe for Microwave use
1	Brown paper bags and newspaper
2	Metal pans
3	Foam insulated cup, bowls, plates or trays
4	Aluminum foil
5	Plastic wraps and cold storage containers

Solvents for Microwave Experiments:

Typically all polar solvents which have OH bonds can absorb microwave radiation. Usually polar solvents includes, water, acids, alcohols, and amides. The polarities of these compounds are in the following order:

Water > Acids > Amides > Alcohols

S. No:	List of Solvents
1	Water
2	Acetic acid
3	Ethanol
4	Isopropanol
5	Formic Acid
6	Acetonitrile

Experiment 1: Comparison of Heating Efficiency by Microwave and Electrical Hotplate

Introduction

The typical methods of heating in chemistry laboratories involve the use of electric hot plates, Bunsen burners and heating ovens. Depending upon the application, microwave heating can be used to replace these conventional methods. Microwaves works by directly coupling with the polar molecules in a substance. This causes the particles to move and rotate which in turn generates heat. Microwave ovens operate with electromagnetic non ionizing radiation with frequencies between 300 GHz and 300 MHz. The corresponding wavelengths span a range from 1 mm to 1 m, which places microwaves in-between that of infrared radiation and radio waves. Most commercial microwave systems however utilize irradiation with a frequency of 2450 MHz (wavelength $\lambda = 0.122$ m) in order to avoid interferences with telecommunication devices. The corresponding electric fields oscillate 4.9 X 109 times per second and consequently subject dipolar species and ionic particles (as well as holes and electrons in semiconductors or metals) to perpetual reorientation cycles. This strong agitation leads to a fast non-contact heating that is (approximately) more or less uniform throughout the radiation chamber. Consequently, faster heating rates are expected in microwaves.

In order to give more emphasis on the energy aspect of these experiments, it is important to compute the actual power consumption in both conventional and microwave heating methods. Every heating system has in-built temperature sensors that turn on/off according to the temperature control set in it. Therefore, the heating device consumes energy only when the power is on. A simple calculation that uses the product of power rating and time led to erroneous results. An inexpensive solution that could be employed is a commercial power meters that can be connected online to the heating device or the microwave. The one selected for this project is called **P3 Kill a Watt** (P3 International Corporation, NY, USA) power meter, which is described below.

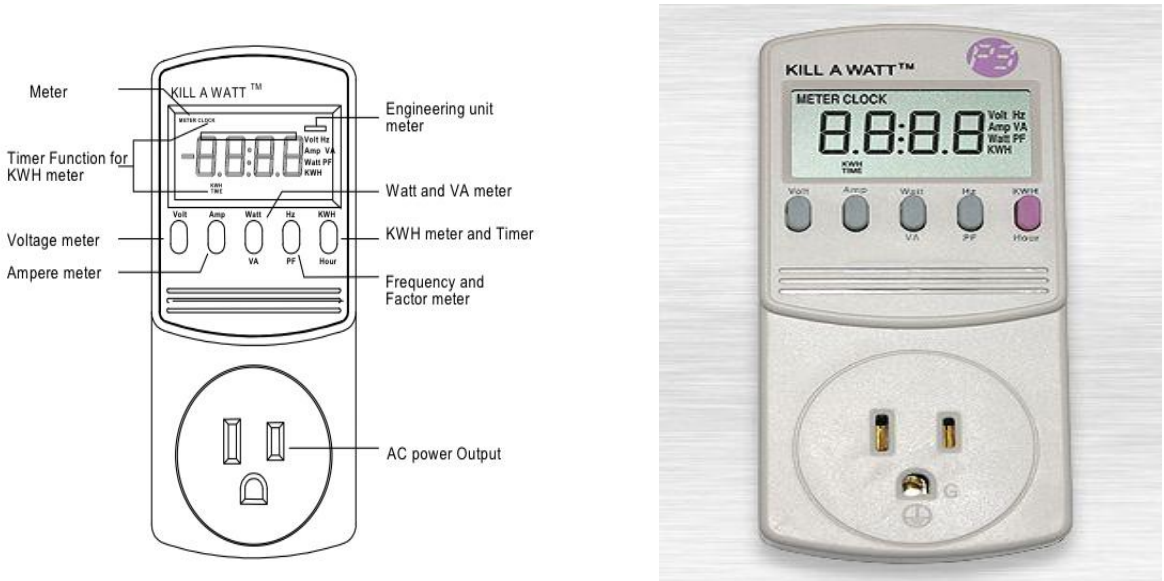


Figure 1. Schematic diagram of Kill a Watt power meter.

P3 Kill a Watt power meter is shown in Figure 1. It is a meter that is used for measuring the power consumption of house hold appliances. This device records six parameters which including voltage, current, wattage, frequency and energy consumption. The measurements are displayed on an LCD screen. The power meter is connected to the main power supply unit while the outlet of the appliance is connected to the power meter. The total power consumed over a period of time is measured in units of Kilo Watt hour (KWH). Both volts and amperes are measured using true RMS method.

Materials Required

- Electric hot plate
- Kill-a-Watt meter
- Microwave
- 1000 ml beaker
- Thermometer
- Stopwatch
- Water

Experimental Procedure

The efficiency of the two methods was compared by using 1000 ml of water in two separate 1000 ml glass beakers and heated by microwave and electric hotplate for different periods of time. The power rating on the hot plate was 865 Watts (51.9 KJ/min) and that of microwave oven was 850 Watts (51 KJ/min). The rise in temperature as a function of time was recorded in each case. The experimental setup is shown in Figure 2. In another set of experiments the effect of volume was computed as follows. Hundred ml volume of water was initially heated for a period of 3 minutes using hot plate. Different volumes in the increments of 100ml were heated for 3mins and the final temperature in each case was recorded. The experiment was repeated with microwave heating, and the corresponding temperatures were recorded.

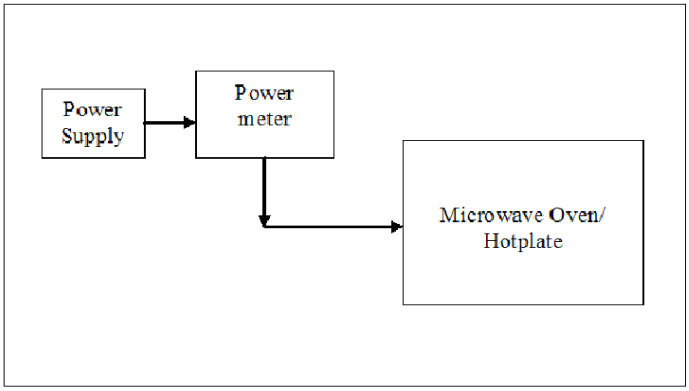
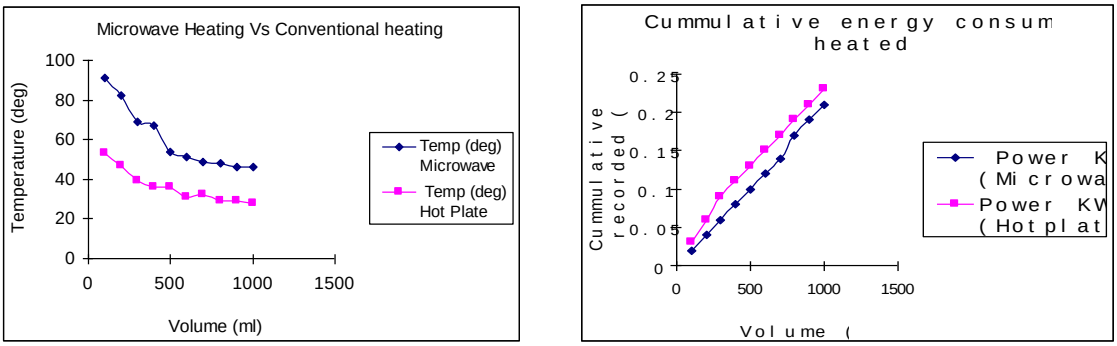


Figure 2 Experimental Setup along with the power meter.

Results and Discussions

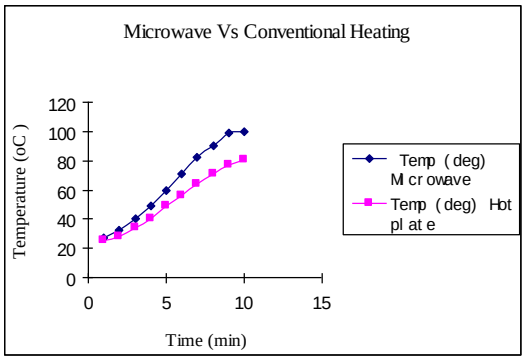
In the first set of experiments, the variation in temperature as a function of volume of water is presented. Figure 3 (a) is the temperature of the water as a function of volume. The temperature decreased significantly as the volume of water increased in both convective and microwave heating. As the volume of water increased, it consumed more energy to raise the temperature. We observed significant decrease in temperature with convective heating, as heat transfer limitations are more prominent with convective heating. For the same volume of water heated for same duration of time, we could see a temperature difference between 20-40˚C, with microwave heating being significantly more efficient. Figure 3(b) shows the power consumed for heating same volume of water for similar duration of time by the two heating methods. The trends were similar, but the absolute numbers were different for the two devices although their power ratings were very close. The microwave consumed less energy but reached higher temperature, which clearly demonstrated the higher efficiency of this approach.



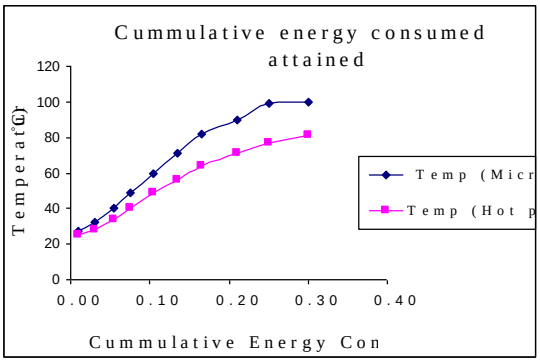
(a) (b)

Figure 3. (a) Plot of temperature as a function of volume at aconstant time duration of 3 mins. (b) Plot of energy consumed as a function of volume for a heating time of 3mins.

Figure 4 (a) shows the temperature of water as a function of time at a constant volume of 1000 ml. During microwave heating, the water temperature increased steadily and leveled off near the boiling point. The rise in temperature was much more gradual with the hotplate. At high temperatures, the vapor pressure of water was quite high, and some water was lost via evaporation. The rate of increase in temperature with increasing time was clearly higher for the microwave than for heating on the hotplate. Figure 4(b) shows temperature reached as a function of energy consumed by each device. It is quite evident that for the same energy consumption, microwave heating is more energy efficient.



(a)



(b)

Figure 4. (a) Plot of temperature as a function of time for heating 1000ml of water. (b) Plot of temperature as a function of energy corresponding to Figure 4 (a).

Energy Calculations

Table 1. Energy consumed by both heating devices as recorded by power meter:

Heating Device	Time (min)	Power Rating (KJ/min)	Actual Energy Consumed (KJ)
Microwave Oven	3	51	72
Hot Plate	3	51.9	90

Difference in Calculated and Recorded Values:
Energy = Power x Time

Amount of Energy Consumed
Microwave Oven: 51KJ/min x 3min = 155.7 KJ
Hot Plate: 51.9 KJ/min x 3 min = 153 KJ

Therefore,

The percentage of error is for microwave oven: $\frac{(153-72)x100}{153} = 52.9\%$

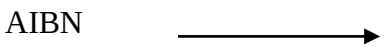
The percentage of error is for hot plate: $\frac{(155.7-90)x100}{155.7} = 42.1\%$

Summary:

- For the same amount of energy consumed by the two heating systems, microwave heating reached higher temperatures.
- As the volume of water increased, more microwave energy was absorbed and overall efficiency increased, the temperature reached by microwave was significantly higher than the convective heating.
- The power meter was important for computing the energy consumption. Direct computation lead to 40-50% error.

Experiment 2: Synthesis of Polyvinylpyrrolidone (PVP)

PVP is a water soluble polymer that also dissolves in other polar solvents. At dry conditions, it is a white blistering powder that absorbs moisture. From a solution it readily forms films which have been employed for coating purposes. PVP is used as binders for the formulation of pharmaceutical tablets, for moistening various personal care products, as food additives, and adhesives, etc. This has also been employed as an excellent blocking agent in Southern blot analysis (molecular biology technique used for detection of specific DNA sequence in DNA samples). PVP is synthesized via a free radical polymerization reaction starting from the vinylpyrrolidone (VP) monomer, using a free radical initiator such as Azobisisobutyronitrile (AIBN).



Vinylprrolidone **Polyvinylpyrrolidone**

Equation 1. Equation for the synthesis of Polypyrrolidone

Materials Required

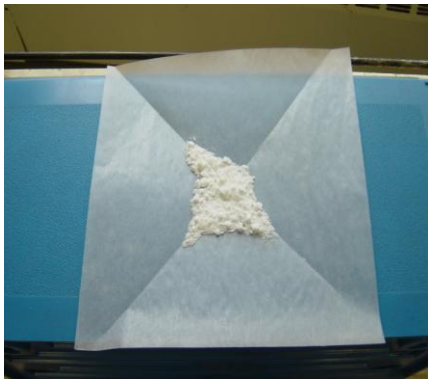
- Two 20 ml bottles with caps
- Two stir bars
- Two volumetric flasks
- Two 100 ml beakers
- Safety goggles
- A set of measuring spoons (reuse)
- A box of weigh paper
- A box of gloves
- A balance
- Two stirring machines
- Towels for cleanup
- One bottle of distillated water

VP and AIBN were purchased from Sigma Aldrich. In comparing the reaction efficiency, these experiments are carried out in a microwave oven and also in a hotplate.

Experimental Procedures

Two bottles (20 ml) containing six to eight mg of free radical initiator, 2,2'-Azobisisobutyronitrile (AIBN), 2 ml vinylpirrolidone (VP) and 2 ml water were stirred for about 20 min at room temperature to dissolve all the AIBN into water. One sample was placed in the microwave oven and the reaction was carried out for 3 minutes and the polymer was formed. The other sample was placed in a hot plate at its maximum power. The polymer was formed in 13 min. The pictures of starting materials and products are presented in Figure 3. The results are presented in the Table below. The net saving in energy was quite evident.

VP (starting material)



AIBN (initiator)

PVP (final product)

Figure 5. Photographs of initial reactants and final product

Result &Discussion

The formation of white jelly-like solids indicated the completion of the polymerization reaction. Once it is allowed to dry, it forms white flakes which readily absorb atmospheric moisture.

Table 2. The energy consumed for the PVP synthesis by different heating methods

Heating Device	Time (min)	Power Rating (KJ/min)	Actual Energy Consumed (KJ)
Microwave Oven	3	51	72
Hot Plate	13	51.9	396

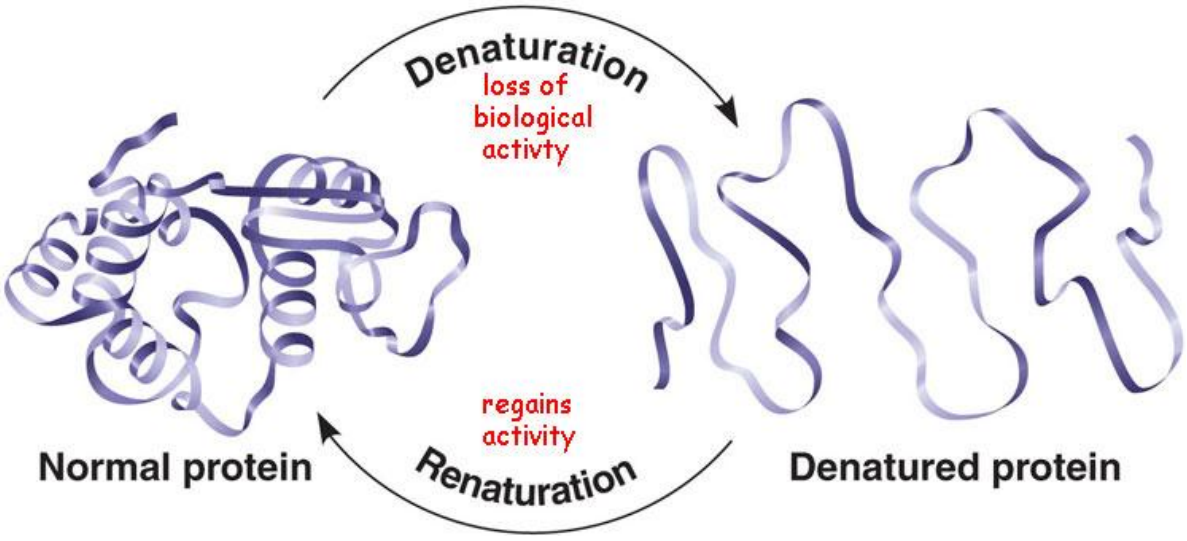
The percentage of energy saved by the microwave oven over the conventional oven as recorded by the power meter,

$$\frac{(396KJ - 72KJ)x100}{396KJ} = 81.8\%$$

Experiment 3: Thermal Denaturization of Protein

Denaturation is the process of modifying the conformation of the protein structures without rupturing the native peptide linkages. This inactivates the functionality of the protein molecules, decreases its solubility, decreases/destroys its biological activity, improves digestibility and alters the water binding ability of the molecule. Denaturation of proteins is achieved by disrupting the hydrogen bonding in the peptide linkage by applying external stress. It can be carried out by applying heat, treatment with alcohols, heavy metals, or acids/bases.

The principle of denaturation has been exploited in the field of food chemistry and in developing genetically engineered compounds. The purpose of denaturing a protein is to inactivate certain components of a living cell, thereby suppressing it from expressing certain properties of the proteins. Protein denaturation is widely used in food processing and dairy industries and a simple example is the cooking of egg white for making an omelets.



* **Figure 6.**A Schematic representation for denaturation of proteins.

* Source: http://www.wikipedia.org

Materials Required

- Two 150ml calibrated glass beakers.
- Two glass rods for stirring.
- Two Eggs.
- Safety goggles.
- Towels for clean up.
- Stop watch.

Experimental Procedure

About 100ml of egg white was separated by slightly cracking the freshly obtained eggs in two separate 150ml beakers. One beaker was placed in the microwave oven and the other on a hotplate. Both specimens were heated to a temperature of 100 ^{o}C. The applied heat caused the breakage of the long peptide bonds of the protein molecules which coagulates as white semi-solid structures. The time taken for coagulation by microwave oven and the hot plate were calculated to be 2 and 14 min respectively. The formation of coagulates are shown in the Figure 7 below.

(a) (b)

Figure 7. (a) Egg white separated from pure egg before coagulation. (b) Egg white after coagulation.

Result & Discussion

The initial material is a viscous liquid. As the heat treatment continues, it coagulates into a whitish material that is semi-solid in nature. The formation of white fluffy solids indicated the completion of the denaturization of the proteins. This material is significantly less insoluble in water than the starting material. The energy consumption by each method is tabulated below.

Table 3: The energy consumed for the protein denaturation by different heating methods

Heating Device	Time (min)	Power Rating (KJ/min)	Energy Consumed (KJ)
Microwave Oven	2	51	72
Hot Plate	14	51.9	288

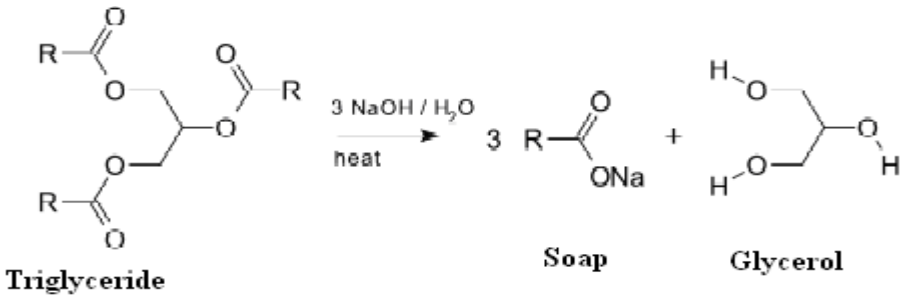
Therefore,
The percentage of energy saved by the microwave oven over the conventional oven as recorded by the power meter,

$$\frac{(288KJ - 72KJ)x100}{288KJ} = 75.0\%$$

From the above experiment, the time taken for denaturation of protein by conventional heating was higher than microwave heating. This is due to the fundamental difference in the heating mechanisms. With microwave heating it was possible to locally heat the target molecules rather than maintaining a whole vessel at an elevated temperature. The former was a more energy-efficient process.

Experiment 4: Saponification of Fat: Synthesis of Soap

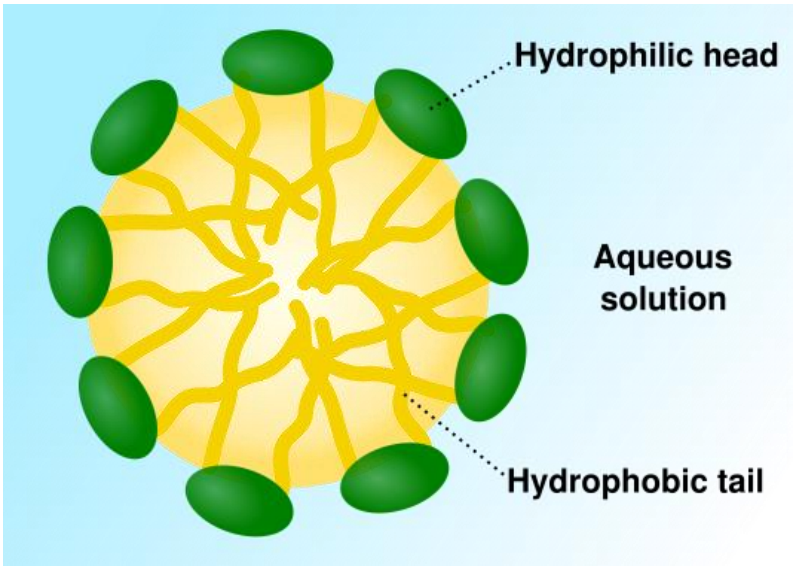
Saponification is the process of making soap from alkali and fat (or oil). Vegetable oils and animal fats are fatty esters in the form of triglycerides. The alkali breaks the ester bond and releases the fatty acid salt and glycerol. If necessary, soaps may be precipitated by salting out with saturated sodium chloride. Usually, sodium hydroxide is used in formation of hard soap while potassium hydroxide is used in case of soft soap.



Equation 2. Saponification Reaction

Mechanism of cleaning by Soap

It is not possible to remove dirt (especially oil and grease) by just using water. The non-polar components present in the dirt repel the polar constituent of the solvent. In presence of a detergent (soap) which has both polar and non-polar ends, the non-polar ends of the detergent which is repelled by water interacts with the nonpolar grease. At the same time, the polar ends are attracted towards the hydrophilic molecules. Thus the two complementary polar and nonpolar components of the dirt are dissolved and removed during washing. The soap forms micelles in water, where the polar ends align along the circumference and non-polar constituents carrying the nonpolar species are remain in the center of the micelle.



*** Figure 8. Typical micelle formation**

*****Source: http://images.google.com

Materials Required

- Animal Fat (Crisco)
- Ethanol
- 6N Sodium Hydroxide
- Sodium Chloride
- Isoproply alcohol
- Two 100 ml Beaker
- Two glass rods
- Two 250ml conical flask
- Distilled water
- Microwave
- Hot plate
- Safety goggles
- Towels for clean up
- Stop watch

Experimental Procedure

Ten grams of commercially available animal fat/shortening was weighed and dissolved in 50 ml of ethanol by constant stirring. To this mixture was added 15ml of freshly prepared 6N sodium hydroxide solution. This mixture was heated on a hot plate until all the fat was completely dissolved. 20ml of distilled water was added and the mixture was cooled on an ice bath. The cooled mixture was then poured into a beaker containing 50ml of 0.2 % Sodium Chloride solution. Soap was formed upon cooling. The solution was filtered to separate the soap from the glycerol thus formed. The experiment was repeated using microwave. The corresponding efficiencies and energy consumptions were calculated.

Result & Discussion

As a result of the saponification process, the fatty acids are hydrolyzed in presence of an alkali so as to form salts of alkali and alcohol. Upon cooling of the dissolved mixture, solid soap was observed the end of the process. Energy consumed by each process is tabulated below.

One way to test the formation of soap is by dissolving the solid in water and checking foam formation. It can also be confirmed by performing pH test, which involves dissolving the soap in a freshly prepared 1:3 water and isopropyl alcohol mixture. The dissolved soap solution can be tested with phenolphthalein indicator and the soap formation is indicated by the color of the solution. A dark pink and clear solution indicates presence of excessive caustic solution, the one with colorless or yellowish clear solution indicates a fairly neutral pH, hazy solution indicates untreated oil, hazy and pink solution indicates the reaction was complete. A clear, pale pink solution indicates good results.

(a) (b) (c)

Figure 9 (a) Unreacted soap solution, (b) Hazy pink solution with unreacted oil, (c) Clear pink solution indicating the formation of soap.

Table 4. The energy consumed for Saponification of 10g of animal fat.

Heating Device	Time (min)	Power Rating (KJ/min)	Actual Energy consumed (KJ)
Microwave Oven	1	51	36
Hot Plate	4	51.9	108

The percentage of energy saved by the microwave oven over the conventional oven as recorded by the power meter,

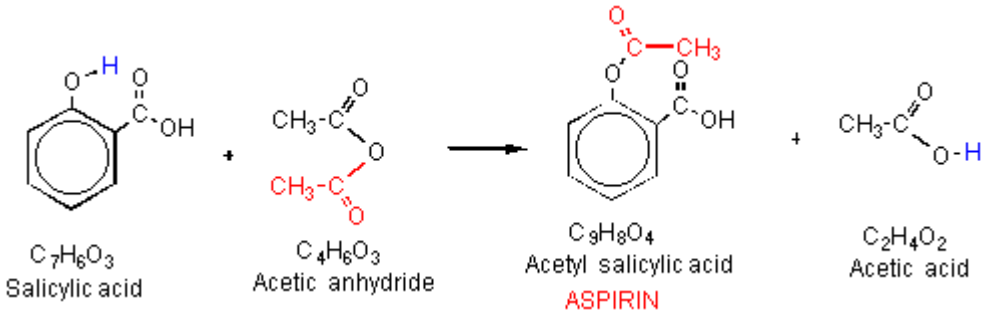
$$\frac{(108KJ - 36KJ)x100}{108KJ} = 94.4\%$$

The saponification process using conventional heating took four times as much time to complete than the microwave process, and consumed more energy. The quality of soap was also tested. From the **Fig. 9 (c)** we see the solution thus formed after the addition of phenolphthalein, was a clear pink solution indicating the formation of good soap with a nominal pH in the range of 7-9. In **Fig 9 (b)** we see a hazy pink solution with yellow droplets in it, indicating the presence of unreacted oil. The solution also appears dark in color indicating the presence of excessive caustic soda. **Fig 9 (a)** shows a clear solution which indicates the absence of soap formation.

The amount of energy consumed by the hotplate was significantly higher than what was consumed by microwave. This was attributed to the direct interaction of the reactants with the microwave radiation.

Experiment 5: Synthesis of Aspirin

Aspirin or acetyl salicylic acid (ASA) is a derivative of salicylic acid, which is used as a pain reliever for various body ailments such as head ache. Aspirin shows anti-platelet or anti- coagulant properties by inhibiting the prostaglandins, thereby repairing damaged blood vessels. Hence aspirin is used in low doses on long term basis in treatment of heart attacks, strokes and in people having high risks for formation of blood clots. The synthesis of acetyl salicylic anhydride from salicylic acid and acetic anhydride is catalyzed by phosphoric acid. The efficiency is estimated by the time taken to complete the reaction.



Equation 3. Reaction equation for the synthesis of Aspirin

Materials Required

- Salicylic acid
- Acetic anhydride
- Phosphoric acid
- Distilled water
- 250 ml Erlenmeyer flask
- Ice cubes
- Hot plate
- Microwave oven
- Stirring rod
- Buchner funnel
- Safety goggles
- Stop watch

Experimental Procedure

18 ml of acetic anhydride was slowly added to 10 grams of salicylic acid in a 250 ml Erlenmeyer flask in the hood. 10 to 20 drops of 85 % phosphoric acid was carefully added to the solution and mixed thoroughly. The mixture was heated on a hot plate until all the salicylic acid was dissolved. Once the reaction was complete, 20 drops of distilled water was cautiously added to the mixture followed by 20 ml of distilled water. The solution was cooled on an ice bath until aspirin crystallized. In event of no crystal formation, the walls of the flask were scratched with a stirring rod to initiate crystallization. The crystals were filterer using a Buchner filter and extracted using chilled water. The solid was then dried in an oven at 100 °C for about 30 minutes, weighed and the yield was calculated. The experiment was

repeated in a microwave oven and the yield was compared. Further analysis could include melting point, IR and NMR measurements.

Figure 10. Represents the final product obtained after synthesis.

Result & Discussion

Solid salicylic acid structures were dissolved in presence of acetic anhydride which is catalyzed by acid. The dissolved mixture on cooling produced white crystals of Aspirin. The corresponding energy consumed by each method of heating is tabulated below.

Table 5. The energy consumed for the synthesis reaction

Heating Device	Time (min)	Power Rating (KJ/min)	Actual Energy Consumed(KJ)	Yield (%)
Microwave Oven	1	51	36	95
Hot Plate	5	51.9	180	90

From the data presented in table 5, the microwave method of synthesis proved to be more efficient than the hotplate. Fig. 10 shows the final aspirin product obtained after synthesis process. The synthesis process was enhanced in the case of microwave method of heating thereby saving energy. The microwave process also yielded a slightly higher amount of product than the hot plate.

The percentage of energy saved by the microwave oven over the conventional oven as recorded by the power meter,

$$\frac{(180KJ - 36KJ)x100}{180KJ} = 80.0\%$$

Experiment 6: Extraction of Iron from Oat Meal

Iron is an important nutrient for plants and animals. Iron is a major component of the pigment called "heme" which acts as oxygen carriers in the human body. Deficiency in iron leads to anemia. The greatest need for iron is during growth or periods of blood loss. Young children, adolescents and pregnant women have increased needs for iron because of the growth processes during these periods. In order to make up for the regular iron loss, iron has to be supplemented in the regular diet through food. The measurement of iron in food stuff is for great importance.

Since iron in most food products is at trace levels, its analysis is relatively complicated. Typically the metal is first extracted using a solvent (acid or chelating agent) and then analyzed. The objective of this experiment is to extract iron from food using conventional method such as hot plate and then compare it to microwave. The experiment involves acid extraction and the estimation of extraction efficiency.

Materials Required

- Oat meal (obtained from supermarket)
- 10 % Nitric acid
- 1% Potassium thiocyanate solution (KSCN)
- Ferric chloride
- Distilled water
- 250 ml glass beaker
- Glass rod
- Graduated cylinder
- Filter paper No.1
- Microwave oven
- Hot plate
- UV-Spectrometer
- Cuvette

Experimental Procedure

1% Potassium Thiocyanate (KSCN) was prepared by dissolving 1.0 g of potassium thiocyanate in 100 ml of 10% nitric acid solution. 10mg/ml Iron (III) Chloride stock solution was prepared by dissolving10g of Iron (III) Chloride Hexahydrate in 1litre of 10% nitric acid solution. Five dilute standards of 0.2, 0.4, 0.6, 0.8, and 1.0 mg/mL were then prepared from the stock solution.

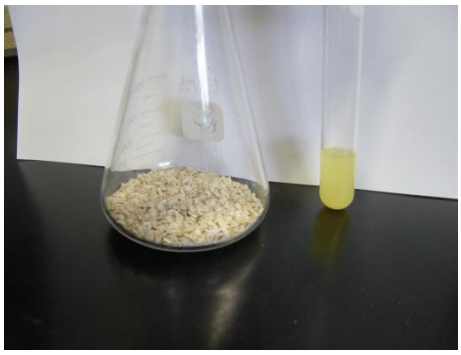
Sample Preparation

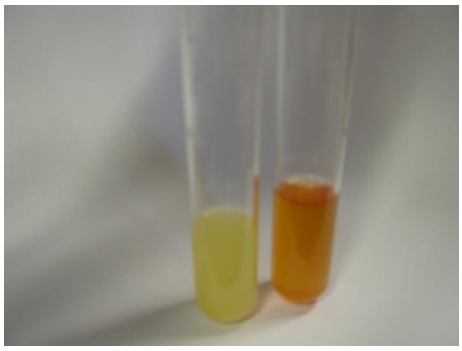
A measured volume of 50 ml of 10% nitric acid solution was added to 10g of oatmeal (from Grocery store) and heated on a hot plate until the oatmeal dissolved. The time taken for the extraction reaction to go to completion was recorded. The solution was then filtered and 0.1ml of 1% KSCN was added to the filtrate forming a blood red colored complex represented by the equation below. The absorbance of the colored complex was measured at 450 nm on a UV spectrometer. The process was repeated in a microwave oven to compare the extraction efficiency. The change in color is show in the figure 11.

$$Fe^{3+}(aq.) + 3SCN^{-}(aq.) \longrightarrow Fe(SCN)^{3}(aq.)$$

Equation 4. Formation of Ferric thiocyanate complex.

A calibration curves was generated by successive dilution of the standard, A mixture of 10% nitric acid and 0.1 mL of 1% KSCN solution was used to dilute the standards, and their absorbance was measured at 450nm on the UV-spectrometer. The 10% nitric acid solution was used as the blank. Absorbance was plotted against the concentration of standards and is presented in figure 12.





(a) (b)

Figure 11. (a) Oatmeal to be extracted; b) Represents the iron extracted from oats before the formation of the color complex. (b) The color change after complex formation.

Results and Discussion

The iron was extracted from oat meal by acid extraction, which dissolved the oat meal on heating. The filtrate separated from the extract formed color complexes with potassium thiocyanate solution, which was analyzed using a UV- Spectrometer.

Figure 12, represents the calibration curve obtained by plotting the absorbance of known concentrations. The concentration of iron extracted from oat meal was computed from the calibration curve. The efficiency of extraction using the microwave oven and conventional heating were compared based on the amount of iron extracted and the time taken for the extraction process. The extraction efficiency from microwave heating was slightly higher than that of hotplate heating.

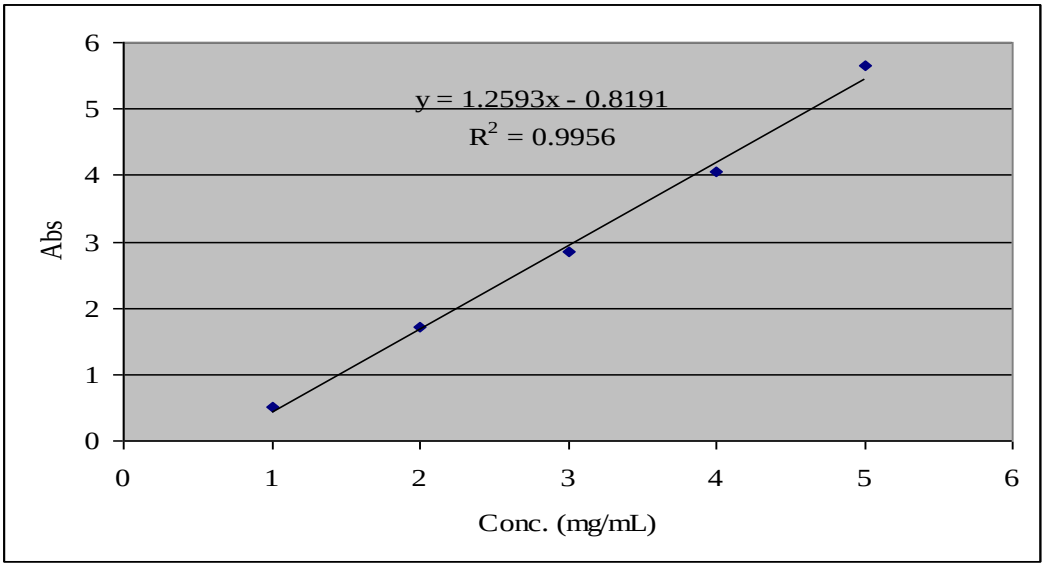


Figure 12. Calibration curve for iron content determination.

Table 6. The energy consumed and the concentration of iron extracted by different heating methods

Heating Device	**Time (min)**	**Power Rating (KJ/min)**	**Actual Energy Consumed (KJ)**	**Amount of Iron present (mg/g)**
Microwave Oven	3	51	108	17.8
Hot Plate	15	51.9	468	17.2

Difference in Calculated and Recorded Values:

Energy = Power x Time

Energy Consumed by each device

Microwave Oven: 51KJ/min x 3min = 153 KJ
Hot Plate : 51.9 KJ/min x 15 min = 778.5 KJ

Therefore,
The percentage of error is for microwave oven,

$$\frac{(153KJ - 108KJ)x100}{153KJ} = 29.4\%$$

The percentage of error is for hot plate,

$$\frac{(778.5KJ - 468KJ)x100}{778.5KJ} = 39.8\%$$

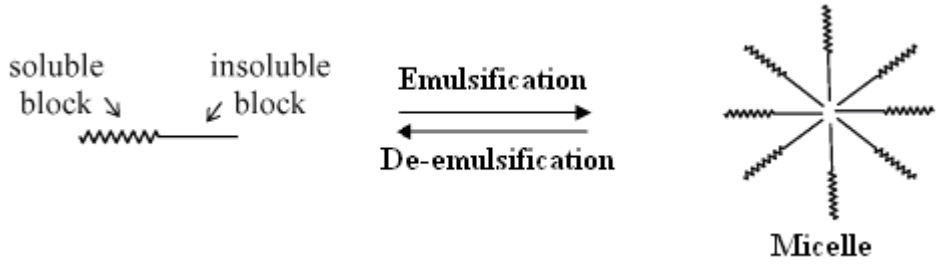
Therefore,
The percentage of energy saved by the microwave oven over the conventional oven as recorded by the power meter,

$$\frac{(468KJ - 108KJ)x100}{468KJ} = 76.92\%$$

From the results it is evident that though the hotplate took five times as much time to complete extraction, a slightly higher concentration of iron was extracted in the microwave process translating to about 76.9 % energy saving. The direct heating capability of microwaves can be credited with the superior heating and extraction efficiency.

Experiment: 7 De-emulsification of Oil by heat

Emulsion is a mixture of two different immiscible liquids, where droplet of one forms stable colloids in the other solvent. The process is enhanced by the addition of an emulsifying agent or a surfactant. De-emulsification is a process where the mixtures of the immiscible liquids are separated. In this experiment, oil and water is taken as the two immiscible liquids which are mixed in different proportions and mixed with a surfactant (generally detergent). Separation of this emulsion is achieved by application of heat. Addition of surfactant causes a micelle formation by hydrophilic and hydrophobic interactions as shown in the figure below. Application of heat causes breakdown of these micelles, interrupting the hydrophilic-hydrophobic interactions. This leads to the separation of the two immiscible liquids. The process of de-emulsification is used in industry for important functions such as the purification of heavy crude oil in petroleum and solvent removal in paint industries.



Equation 5. De-emulsification reactions

Materials Required

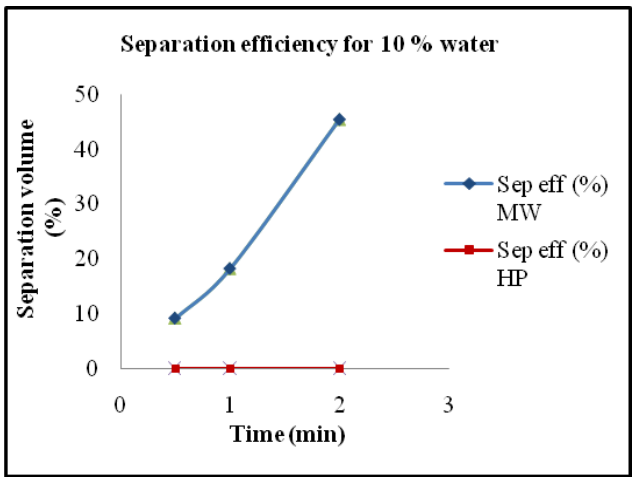
Two 1000ml graduated glass beaker.
Two glass rods for stirring.
Measuring jar
Cooking oil
Distilled water
Sodium dodecyl sulfate (SDS).
Stirring Machine
Safety goggles.
Towels for clean up.
Stop watch.

Experimental Procedure

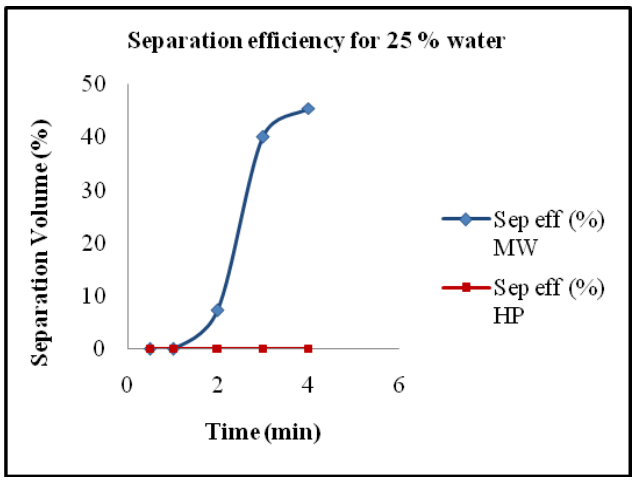
The preparation of stable emulsion can be challenging. We prepared stable oil-water emulsions with Sodium Dodecyl Sulfate (SDS) as the emulsifying agent. Emulsions containing 10%, 25% and 50% water in oil were prepared. The amount of SDS added was proportional to the percentage of water added. In case of 10, 25and 50% of water emulsion, the amount of SDS was 0.05, 0.1 and 0.2 gms respectively. The solutions were heated at for different periods of time to determine the separating efficiency.

Result & Discussion

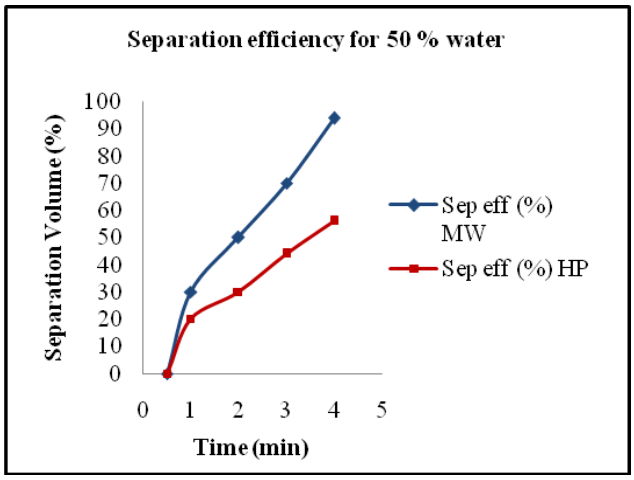
From the Fig 13 (a) and (b), the amount of water separated by hotplate in case of 10 % and 25 % in comparison with microwave. At lower percentage of water, hot plate hardly separated the water from the mixtures. As it required more amount of energy to break down the micelle bonds. In case of microwave radiation, the direct heat applied by it was sufficient enough to break down the strong micelle bonds formed at lower percentage of water content. Higher the percentage of water, more unstable the micelle bond becomes, hence it requires less energy to break down the micelles in order to separate the emulsion. Thus from the Fig 13 (c) & (d) we see that for a 50 % micelle mixture and 75% micelle mixture a substantial amount of water separated by hot plate heating too. But for the same period of time the amount of water separated by microwave was comparatively higher than that of conventional heating.



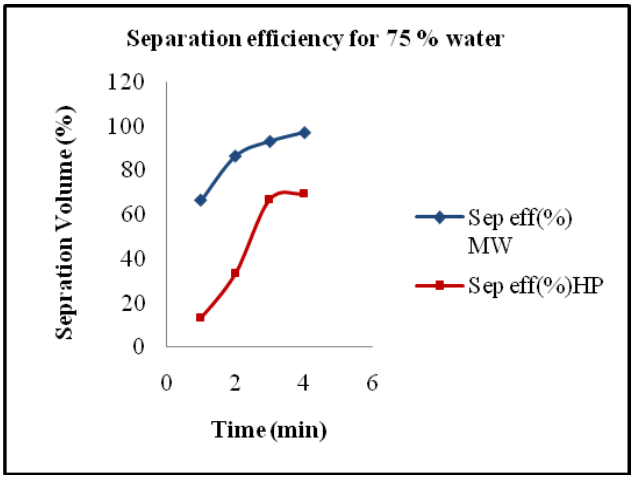
(a)



(b)



(c)



(d)

Figure 13. (a) Volume of water separated versus time for 10 % emulsion mixture, (b) Volume of water separated versus time for 25 % emulsion mixture, (c) Volume of water separated versus time for 50 % emulsion mixture.(d) Volume of water separated from 75 % emulsion mixture.

The amount of water separated by both methods of heating increased with increase in amount of water content.

Table 7. Energy consumed by both heating system for de-emulsification process.

Heating Device	Time(min)	Power Rating (KJ/min)	Actual Energy Consumed (KJ)	Amount of water separated(ml)
Microwave Oven	3	51	72	55
Hot Plate	3	51.9	144	38

Difference in Calculated and Recorded Values:

Energy = Power x Time

Actual Energy Consumed
Microwave Oven: 51KJ/min x 3min = 155.7 KJ
Hot Plate: 51.9 KJ/min x 3 min = 153 KJ

Therefore,
The percentage of error is for microwave oven,

$$\frac{(153KJ - 72KJ)x100}{153KJ} = 52.9\%$$

The percentage of error is for hot plate,

$$\frac{(155.7KJ - 144KJ)x100}{155.7KJ} = 8.12\%$$