Green Chemistry in Teaching Laboratory
Microwave Induced Reactions

by

USEPA & NJIT

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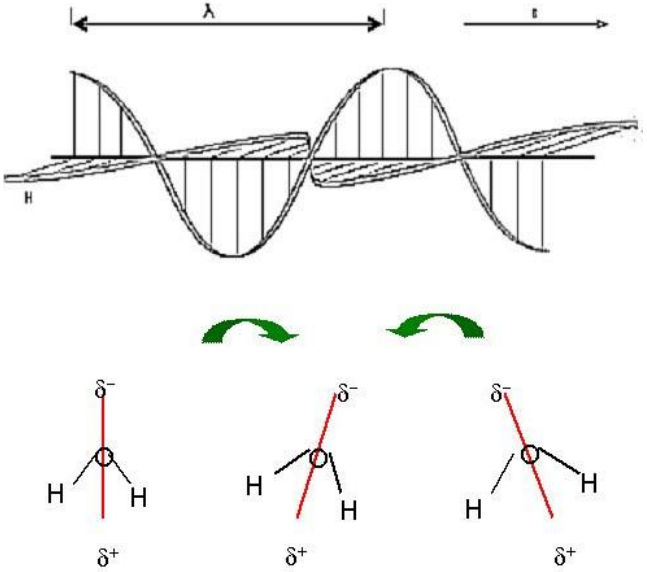
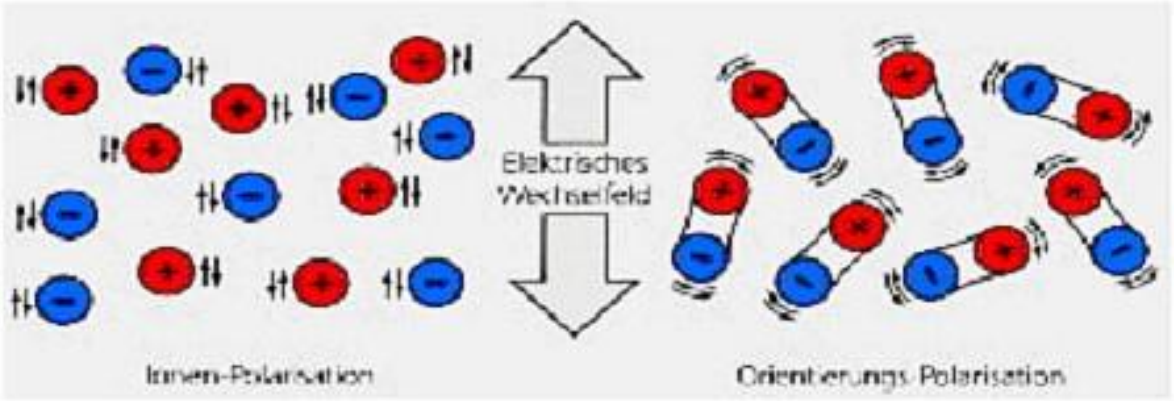
Microwave Heating

- Started domestic use around the mid-1970s.
- Typically for heating food and beverages.
- Uses electromagnetic waves in the Ultra-High, Super-High, and Extremely-High Frequency range (0.3 GHz to 300 GHz)

Principle

- One of two typical ways:
- Dipole Moments
- Ionic Solutions



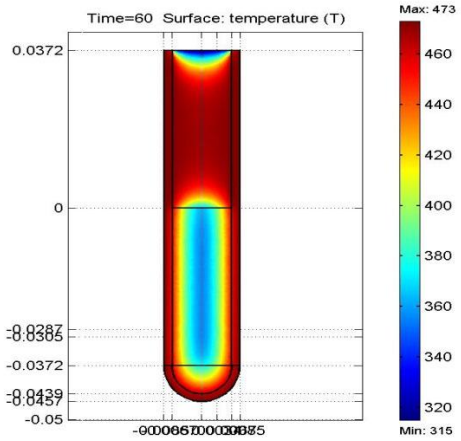


Solvents Used

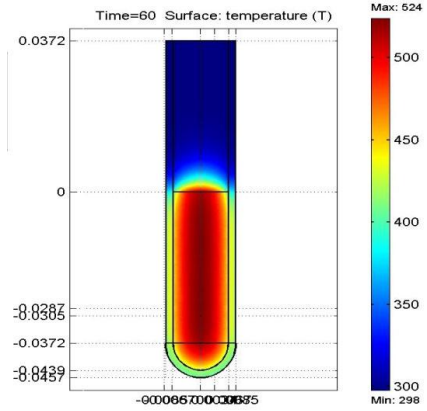
Poor	Medium	Good
Chloroform	Water	Methanol
Dichloromethane	Acetonitrile	Ethanol
CCl_4	Acetone	Propan-2-ol
n-Hexane	Ethylacetate	1-Butanol
Toluene	Tetrahydrofurane	Ethylene glycol
Xylene	DMF	

Conventional & Microwave Heating

- Conventional heating is slower and less efficient.
- Microwave energy heats uniformly and evenly.
- Faster and safer.



Conventional Heating



Microwave Heating

Energy Measurements

- 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 commercial power meter called **P3 Kill a Watt** that can be connected online to the heating device or the microwave to determine the actual power consumption by the system.

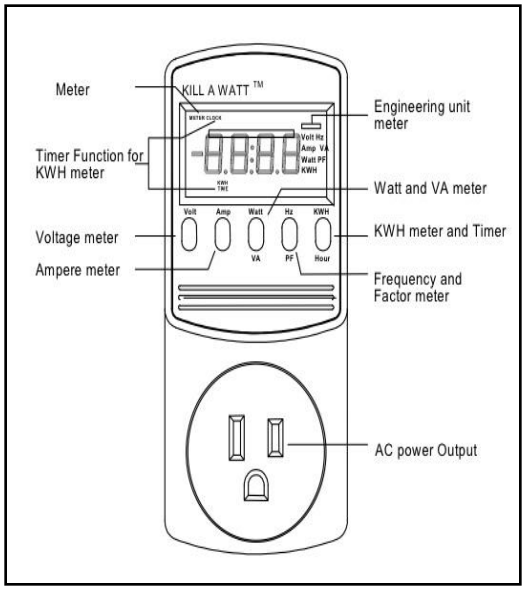


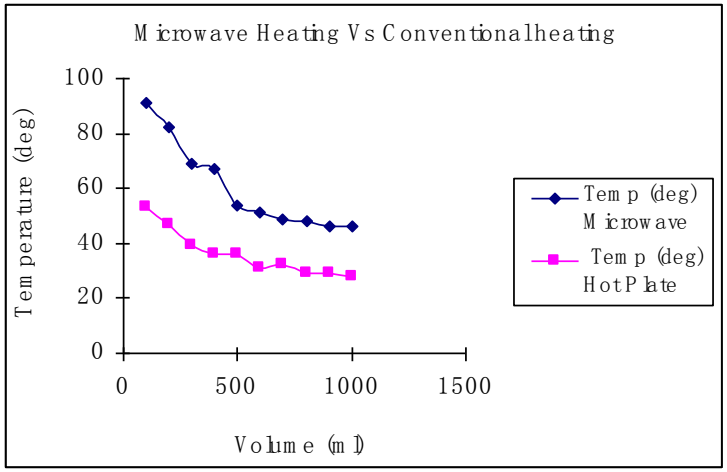
Figure. Schematic diagram of Kill a Watt power meter.

List of Experiments

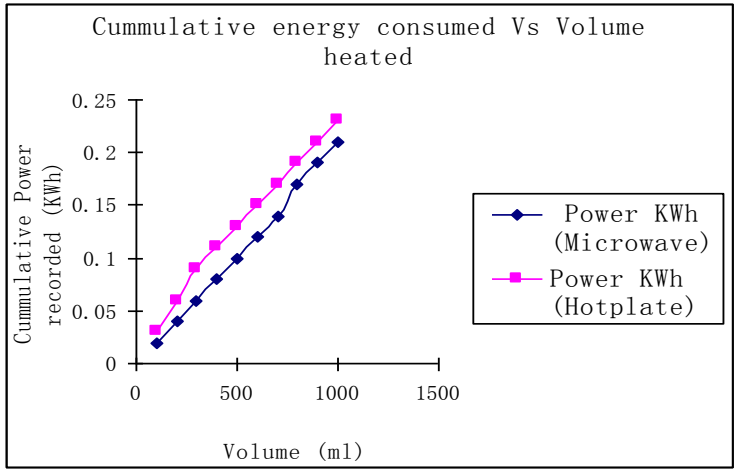
- Comparison of heating efficiency by microwave and hot plate
- Synthesis of Polyvinly propylidone
- Protein denaturation by heat
- Saponification of Fat : Synthesis of Soap
- Synthesis of Aspirin
- Extraction of iron from Oat meal
- De-emulsification of oil by heat

Comparison of Heating Efficiency by Microwave and Hot Plate

- Temperature as a function of volume of water heated was studied.
- Temperature as a function of time for heating water by both methods were studied.
- 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.

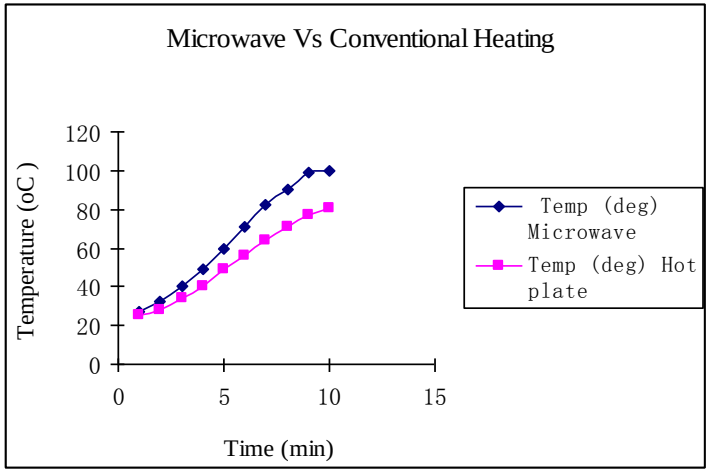


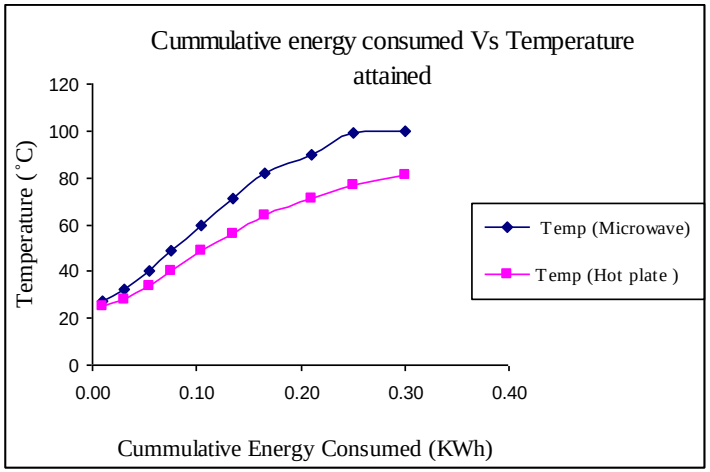
(a)



(b)

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





(a) **(b)**

Figure. (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

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: $\frac{(155.7-90)x100}{155.7} = 42.1\%$

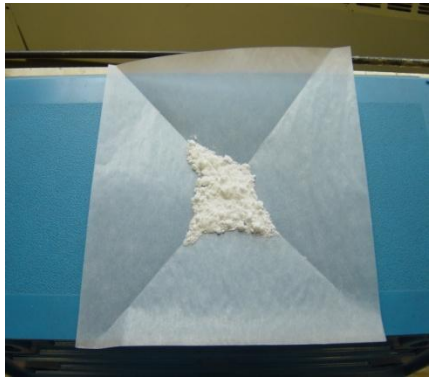
Synthesis of PVP

- 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.

Vinylprrolidone **Polyvinylpyrrolidone**

Equation for the synthesis of Polypyrrolidone

Vinyl pirrolidone
(starting material)



2,2’-Azobisisobutyronitrile
(Initiator)

Polyvinylpyrrolidone
(Final Product)

Figure. Photographs of initial reactants and final product

Thermal Denaturization of Protein

- Denaturation is the process of modifying the conformation of the protein structures without rupturing the native peptide linkages.
- 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.

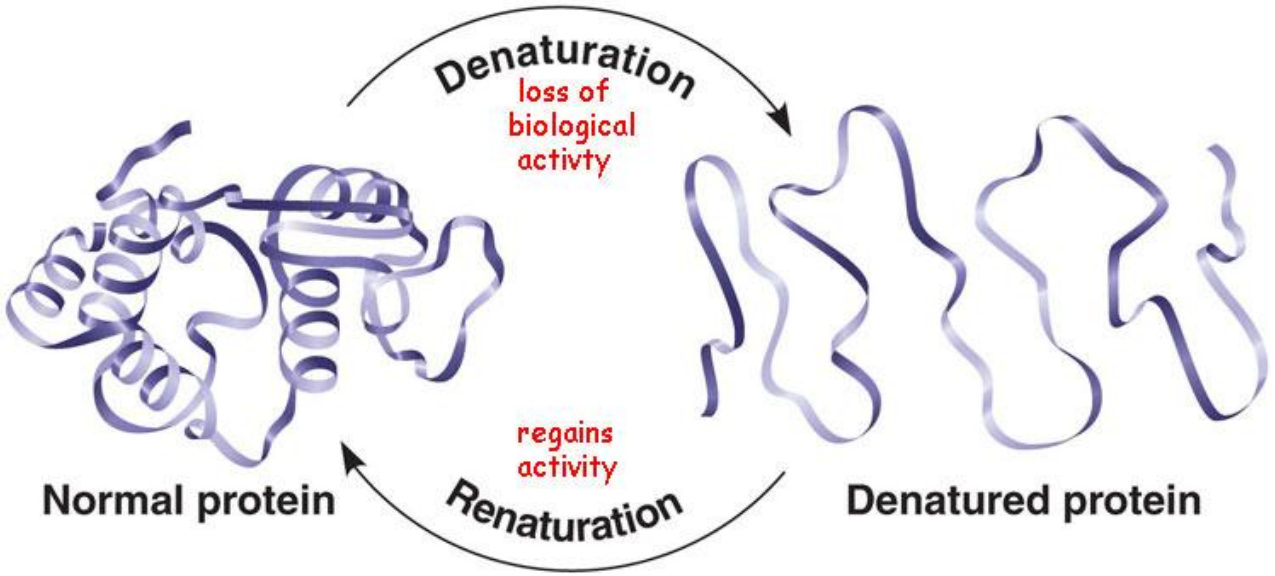
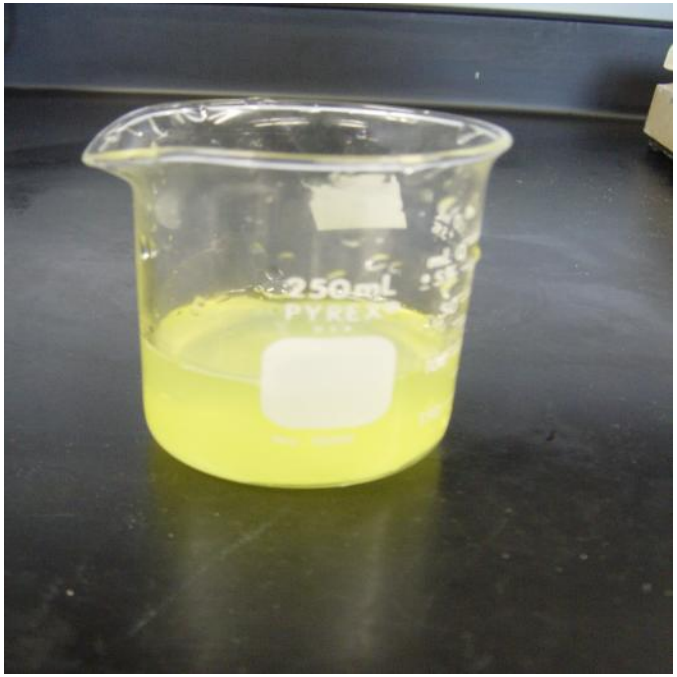
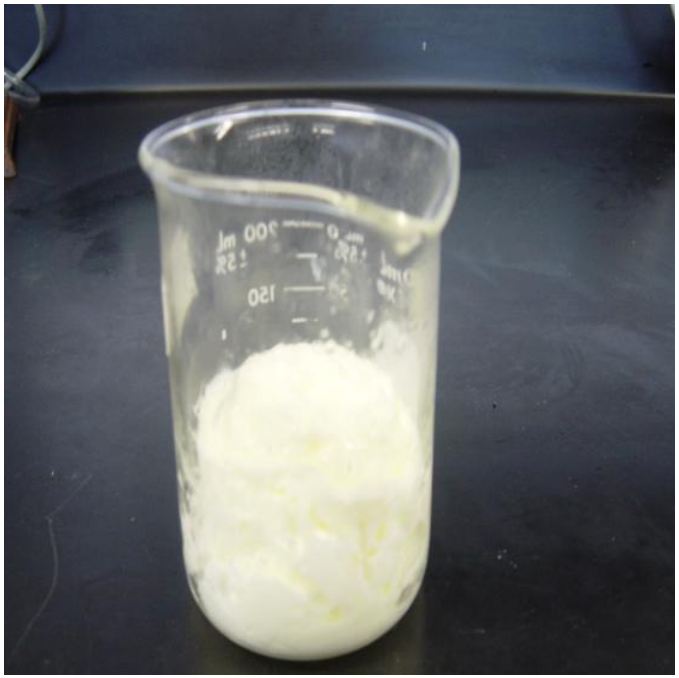


Figure. A Schematic representation for denaturation of proteins



(a)

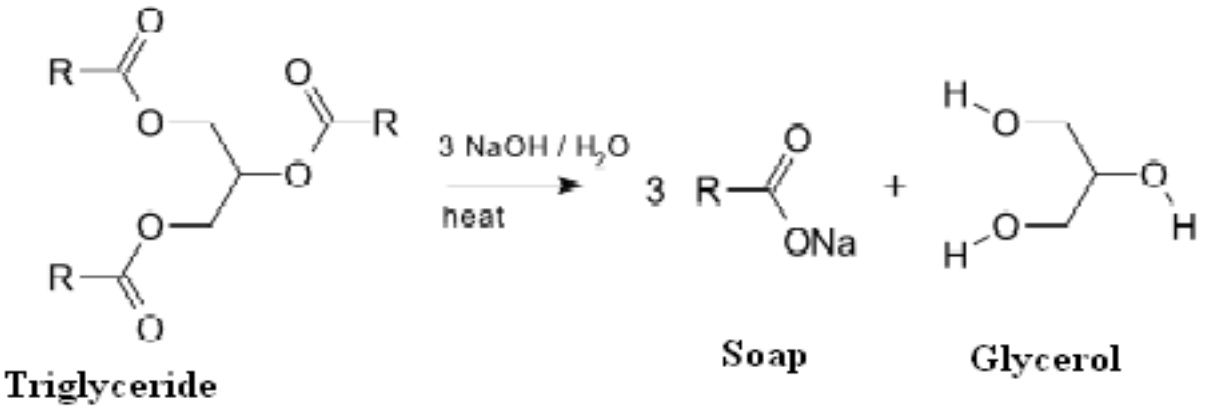


(b)

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

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.



Equation . Saponification Reaction

Table. Energy calculation for saponification process

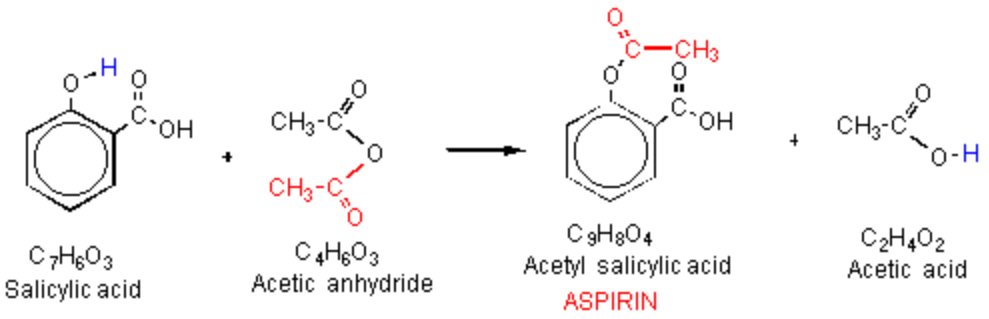
Heating Device	**Time (min)**	**Power Rating (KJ/min)**	**Actual Energy consumed (KJ)**
Microwave Oven	1	51	36
Hot Plate	4	51.9	108

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

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

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.
- The synthesis of acetyl salicylic anhydride from salicylic acid and acetic anhydride is catalyzed by phosphoric acid.



Equation. Reaction equation for the synthesis of Aspirin

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

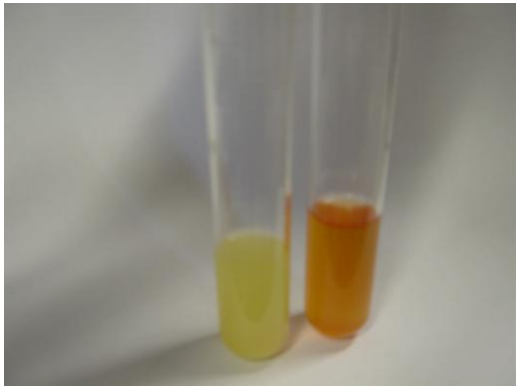
The percentage of energy saved by the microwave oven over the conventional oven as recorded by the power meter,

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

Extraction of Iron from Oat Meal

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.

(a)

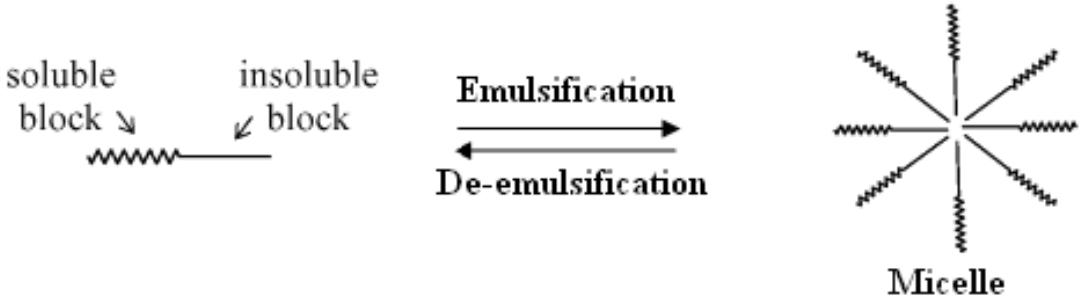


(b)

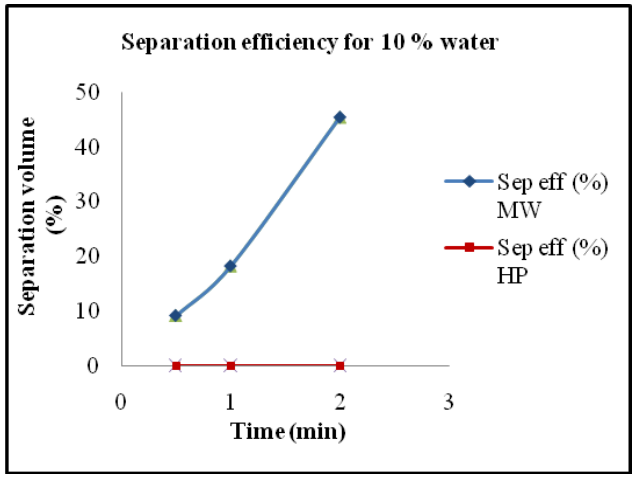
Figure. (a) Oatmeal to be extracted; b) Represents the iron extracted from oats before the formation of the color complex. (b) The color change after complex formation

De-emulsification of Oil by heat

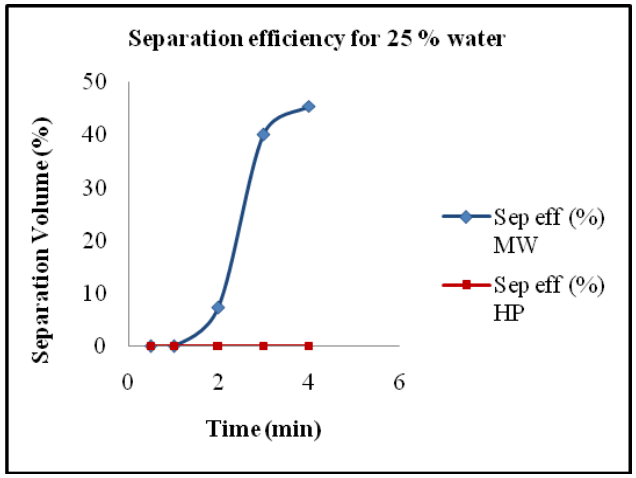
- De-emulsification is a process where the mixtures of the immiscible liquids are separated.
- In this experiment, oil and water is taken as the two immiscible liquids which are mixed in different proportions and mixed with a surfactant (generally detergent).
- Separation of this emulsion is achieved by application of heat.



Equation. De-emulsification reactions



(a)



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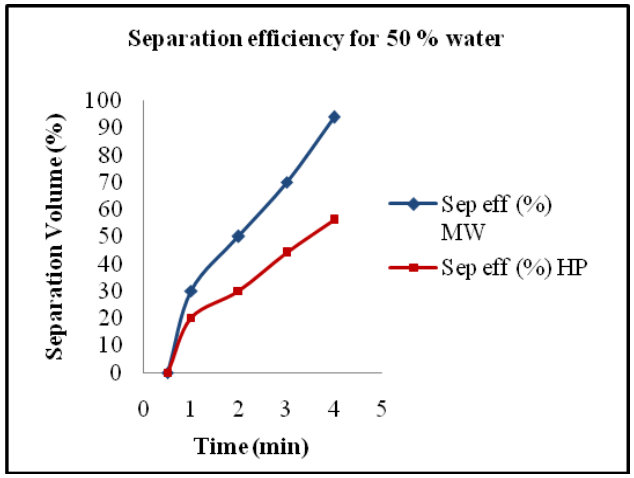

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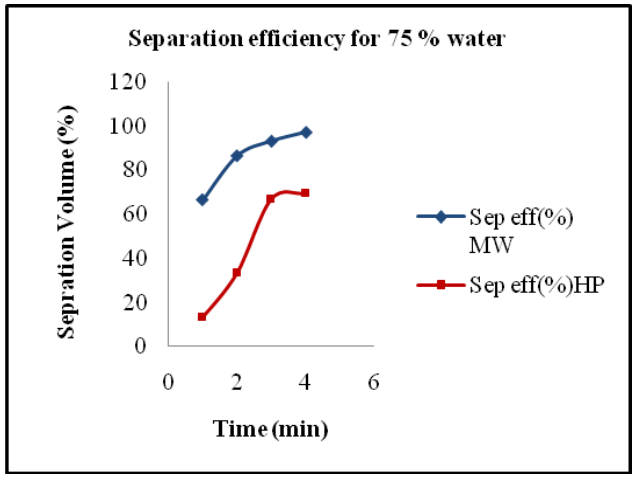

(d)

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.