Introduction to Microwave Heating

Introduction

A microwave is a low energy electromagnetic wave with a wavelength in the range of 0.001–0.3 meters (**Figure 1**). Though microwaves are commonly associated with reheating leftover food, they play important roles in other applications, such as heating laboratory experiments.

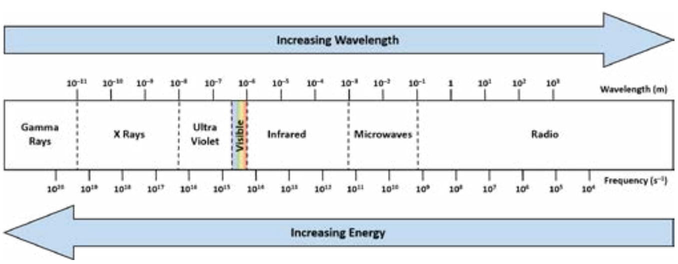

Figure 1: The electromagnetic spectrum

A microwave, like any other electromagnetic wave, consists of two perpendicular oscillating fields: an electric field and a magnetic field. In the case of microwaves, the electric field is primarily responsible for generation of heat, interacting with molecules via two modes of action: dipolar rotation and ionic conduction (**Figure 2**).

In dipolar rotation, a molecule rotates back and forth constantly to align its dipole with the ever-oscillating electric field; the friction between each rotating molecule results in heat generation.

In ionic conduction, a free ion or ionic species moves translationally through space to align with the changing electric field. Like in dipolar rotation, the friction between these moving species results in heat generation, and the higher the temperature of the reaction mixture, the more efficient the transfer of energy becomes. In both cases, the more polar and/or ionic a species, the more efficient rate of heat generation.

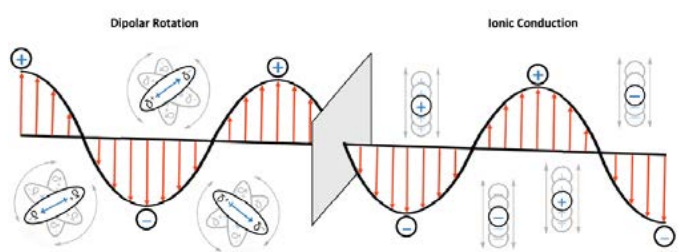

Figure 2: Mechanisms of microwave heating: dipolar rotation and ionic conduction

Because microwaves interact directly with the contents of a reaction mixture, energy transfer occurs more efficiently than with conventional heating techniques. Conventional heating techniques rely on thermal conductivity, where heat is transferred first from source to vessel, and then from vessel to solution. Microwaves interact with a solution uniformly, heating evenly and quantitatively (**Figure 3**).

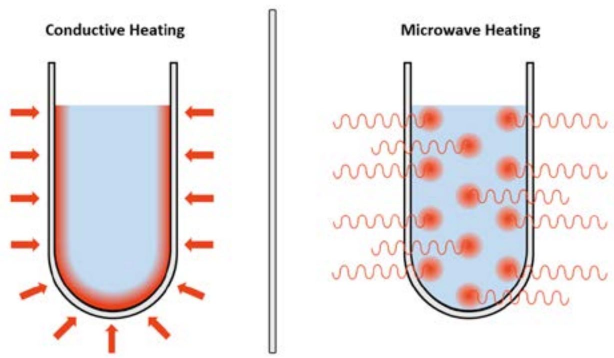

Figure 3: Approaches to heating: conductive heating and microwave heating

Through a series of experiments (outlined below), the principles of microwave heating will be explored.

Materials and Methods

Reagents

Benzaldehyde, deionized water, ethanol, hexanes, iron(II, III) oxide (Fe_3O_4), saturated aqueous sodium chloride (brine), and trihexyltetradecylphosphonium chloride (THTDP Cl).

Procedure

Part I: Ramp to Temperature

1. Program a one-step "Dynamic" method. Method parameters include: (maximum) Temperature = 150 °C, (maximum) Power = 300 W, (maximum) Pressure = 300 psi, Ramp Time = 1 min, Hold Time = 30 sec, PowerMax = Off, and Stirring = High.

   Note: If Synergy software is not being used, Program a "Standard" method on the Discover SP™.
2. Charge a 10-mL vessel, equipped with stir bar, with the indicated volume of solvent or solution (**Table 1**).
3. Seal the vessel with a Teflon-lined silicone cap and placed in the Discover SP microwave cavity.
4. Begin the heating method by pressing the Play button.

   Note: If Synergy software is not being used, record the displayed temperature and power every 10 seconds.
5. Allow the reaction vessel to cool below 50 °C and remove from the Discover SP microwave cavity. Empty the vessel contents into an appropriate waste container and dispose of the Teflon-lined silicone cap.
6. Repeat this procedure for each entry in **Table 1**.
7. Graph the temperature and power data. Using the Synergy software, right-click on the data file and select "Save to File", saving the files as a .csv format and graph.

   Note: If data was manually collected, enter the temperature and power data into a .csv file and graph.

Table 1: Ramp to temperature experiments

Entry	Reagent 1	Reagent 2
1	Deionized water (2.00 mL)	N/A
2	Brine (2.00 mL)	N/A
3	Ethanol (2.00 mL)	N/A
4	Hexanes (2.00 mL)	N/A
5a	Hexanes (2.00 mL)	$Fe30_4$ (0.100 g)
6a	Hexanes (2.00 mL)	Benzaldehyde (1.00 mL)
7a	Hexanes (2.00 mL)	THTDP Cl (0.50 mL)

[a]Ensure that the solution is homogeneous before beginning the heating method.

Part II: Fixed Power

1. Program a one-step "Fixed Power" method. Method parameters include: Power = 300 W, (maximum) Temperature = 150 °C, (maximum) Pressure = 300 psi, Hold Time = 30 sec, PowerMax = Off, and Stirring = High.
2. Charge a 10-mL vessel, equipped with stir bar, with the indicated volume of solvent or solution (**Table 2**).
3. Seal the vessel with a Teflon-lined silicone cap and placed in the Discover SP microwave cavity.
4. Begin the heating method by pressing the Play button.

   Note: If Synergy software is not being used, record the displayed temperature and power every 10 seconds.
5. Allow the reaction vessel to cool below 50 °C and remove from the Discover SP microwave cavity. Empty the vessel contents into an appropriate waste container and dispose of the Teflon-lined silicone cap.
6. Repeat this procedure for each entry in **Table 2**.
7. Graph the temperature data for all entries on the same chart. Using the Synergy software, right-click on the data file and select "Save to File", saving the files as a .csv format and graph.

   Note: If data was manually collected, enter the time and temperature into a .csv file and graph.

Table 2: Fixed power experiments

Entry	Reagent 1	Reagent 2
1	Ethanol (2.00 mL)	N/A
2	Hexanes (2.00 mL)	N/A
3a	Hexanes (2.00 mL)	$Fe30_4$ (0.200 g)
4a	Hexanes (2.00 mL)	Benzaldehyde (1.00 mL)

[a]Ensure that the solution is homogeneous before beginning the heating method.

Discussion Questions

1. Compare the following graphs for the Ramp to temperature experiments. Comment on which sample heats better (requires less power), and why.
2. What mechanism of microwave heating occurs in each sample?
    a. water vs. brine
    b. hexanes vs. ethanol
    c. hexanes vs. hexanes + Fe_3O_4
    d. hexanes vs. hexanes + benzaldehyde
    e. hexanes vs. hexanes + THTDP Cl
3. Compare the graphs for the Fixed Power experiments, all plotted on the same graph. Can you make a true statement about with molecule better absorbs microwave energy? Why or why not?

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