Microwave Frequency Effect(s) in Organic Chemistry

Satoshi Horikoshi[a],* and Nick Serpone[b]

[a]*Department of Materials and Life Sciences, Faculty of Science and Technology, Sophia University, 7-1 Kioicho, Chiyodaku, Tokyo #102-8554, Japan*

[b]*Gruppo Fotochimico, Dipartimento di Chimica Organica, Universita di Pavia, Via Taramelli 10, Pavia, 27100, Italy*

Abstract: This brief article reviews the behavior of microwaves in organic syntheses from the viewpoint of the frequency effect, which has been examined on various common solvents with a newly fabricated 5.80-GHz microwave organic synthesis apparatus, whose features are compared with a similar 2.45-GHz microwave apparatus. Results from usage of the 5.80-GHz microwaves are also compared to the more frequently used MW frequency of 2.45 GHz. The frequency effect was examined for various organic reactions such as the Diels-Alder reaction, the synthesis of benzimidazole-based room-temperature ionic liquids, and the Suzuki-Miyaura coupling reaction. Non-polar solvents can prove particularly useful in organic reactions with the higher frequency microwaves. In this regard, further experiments on microwave-assisted organic syntheses will extend our understanding of the microwave frequency effect(s).

Keywords: Microwave organic synthesis, Frequency effect, 5.8 GHz, Ionic liquid, Suzuki-Miyaura coupling reaction.

INTRODUCTION

Two rather familiar devices that make extensive use of this low energy radiation are the microwave cooking oven and the cellular phone. Early (since 1949) industrial examples include the thermal molding of wood and plastics, the drying of medicinal products, fibers, teas, and cigarettes. In recent years, microwaves have also been used in cancer treatments (hyperthermia), in drying and sterilizing foodstuffs, and in vulcanizing rubber, among others. In chemical and material sciences, fundamental research with microwaves in the mid 1970s focused on microwave heating inorganic materials, with some active research in the early 1980s being directed at the microwave sintering of ceramics [1] in which the principal feature was the formation of compact crystal grains in short time at relatively low temperatures. In the organic field, microwave irradiation to drive organic syntheses was not explored until the mid-1980s, i.e. not until the first two reports in 1986 by Gedye *et al.* [2] and Giguere and coworkers [3] on microwave enhanced organic syntheses. Since then, many organic chemists have discovered the benefits of microwaves to drive synthetic reactions, as a consequence of which industries began to manufacture microwave ovens specifically designed for research laboratories. In this regard, the number of reports on the use of microwaves as an energy source to assist chemical reactions has witnessed an astronomical growth since the earlier decades [4].

Conversion of Microwave Energy to Heat

Microwave radiation represents electromagnetic radiation that spans the frequency range 30 GHz to 300 MHz; i.e., it spans the range of wavelengths from 1 m to 1 cm. Such wavelengths impart molecular behaviors different from those when molecules are subjected to shorter wavelength radiation (e.g. UV, visible and infrared). Phenomena such as vibrations of electrons (electron polarization) and molecules (ion polarization) are commonplace on molecules exposed to various short wavelength ranges of the electromagnetic radiation. By contrast, radio waves with wavelengths longer than infrared give rise to molecular alignment (i.e. alignment polarization).

Electron polarization and ion polarization represent transformations of the electron cloud and the phenomenon of relative displacement of ions in a crystal, respectively, which are caused by the microwaves' electric field. These polarizations are generated regardless of the polar or non-polar nature of the molecules. By contrast, alignment polarization occurs only when the polarity of molecules is affected by the electric field because of their permanent dipoles. Thus, microwaves (and radio waves) are simply not just a source of heat. With regard to the latter, three types of heating phenomena caused by microwaves are worth noting: conduction loss heating, dielectric heating, and magnetic loss heating. The thermal energy P produced per unit volume from the conversion of microwave radiation can be estimated from eqn 1, where $|E|$ and $|H|$

$$P = \frac{1}{2}\sigma\left|E\right|^2 + \pi f \varepsilon_0 \varepsilon''_r \left|E\right|^2 + \pi f \mu_0 \mu''_r \left|H\right|^2 \tag{1}$$

denote the strength of the microwaves' electric and magnetic fields, respectively; σ is the electrical conductivity; f is the frequency of the microwaves; ε_0 is the permittivity in vacuum; ε'' is the dielectric loss factor; μ_0 is the magnetic permeability in vacuum; and μ'' is the magnetic loss.

The first term in eqn. **1** expresses conduction loss heating; the second term denotes dielectric loss heating, and magnetic loss heating is given by the third term. Microwave heating of solutions is due mostly to the dielectric loss factor, whereas conduction loss heating involves mostly, albeit not exclusively solid materials (see below). Generation of heat by the magnetic loss mechanism is expected only in magnetic (solid) materials. In this regard, a catalyzed reaction that takes place on a solid catalyst is expected to show characteristic differences relative to the solution bulk because the reacting substrates in the liquid medium may be adsorbed on the solid's surface. Temperature rise in systems involving solely gaseous molecules is negligible because the molecular density is rather small.

Conduction Loss Heating

Taking the hydroxide ion as a typical ionic species with both ionic and dipolar characteristics, conductive loss effects can become larger than dipolar relaxation in solutions that contain large quantities of ionic species. Such losses tend to be slight at ambient temperatures in the case of solids but can change substantially with increase in temperature. A typical example is alumina (Al_2O_3) whose dielectric losses are negligibly small (~10^{-3}) at ambient temperature but can reach fusion levels in a matter of minutes in a microwave cavity. This effect originates from a strong increase of conduction losses associated with the thermal activation of electrons as they migrate from the oxygen $2p$ valence band to the aluminum's $3s3p$ conduction band. In addition, conduction losses in solids tend to be enhanced by defects in materials, which sharply reduce the energy needed to generate electrons and holes in the conduction and valence bands, respectively. In the case of carbon black powder, conduction losses tend to be rather high, which makes this material an attractive impurity additive to induce losses within solids with too small dielectric losses.

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*Address correspondence to this author at the Department of Materials and Life Sciences, Faculty of Science and Technology, Sophia University 7-1 Kioicho, Chiyodaku, Tokyo #102-8554, Japan; Tel/Fax: (+81) 03-3238-4662;
E-mail: horikosi@sophia.ac.jp

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Dielectric Loss Heating

Non-conductive materials are heated by the microwaves' electric field through molecular rotation of the molecules' electric dipoles, as the latter align themselves with the field. With the field alternating, the molecular dipoles reverse direction (vectorially) and accelerate the motion of individual molecules (or atoms), generating heat through molecular friction as they rotate against each other. Dielectric losses can thus heat solutions with polar substrates. By contrast, heating electro-conductive solids (e.g. metals) implicate ohmic losses in the material as a result of the current flow induced by the oscillating electric field.

Magnetic Loss Heating

Magnetic losses occur in the microwave region for metal oxides such as ferrites and other magnetic materials. Such losses are different from hysteresis or eddy current losses because they are induced by domain wall and electron-spin resonance. For optimal absorption of the microwave energy these materials are best positioned at locations of maximal magnetic field. Transition metal oxides possess high magnetic losses and so can be used as added loss impurity additives to induce losses within solids for which the dielectric loss is too small. For instance, induction heating caused by the microwaves' magnetic field occurs in catalyzed reactions that involve solids, an example being the rapid induction heating of magnetite (Fe_3O_4); this excludes hematite (Fe_2O_3) a non-magnetic material [5].

Dielectric loss heating is the main heating process in microwave-assisted organic syntheses. Accordingly, the dielectric loss factor of organic substrates is an important factor that impacts the speed with which a material is heated by the commonly used 2.45-GHz microwaves. In early studies, polar solvents such as the ethylene glycols were chiefly used in organic syntheses. Recent years, however, have shown that microwave-enhanced organic syntheses can also be carried out in poor microwave absorbing solvents by simple addition of highly microwave absorbing SiC pellets [6] or else in the presence of a small quantity of an ionic liquid [7] both of which act as heating aids in such cases.

Penetration Depth of Microwaves

Microwaves can penetrate deep in dielectric loss liquid systems. For example, in low loss dielectrics where $\varepsilon''/\varepsilon' << 1$ and ε' is the dielectric constant, the depth of penetration of microwaves (D_p, in cm) into the absorbing media can be estimated from eqn. **2**, where

$$D_p = \left(\frac{\lambda_o}{2\pi}\right)\left(\frac{\sqrt{\varepsilon'}}{\varepsilon''}\right) \qquad (2)$$

λ_o is the wavelength of the irradiation ($\lambda_{o(2.45\text{-GHz})} = 0.122$ m; $\lambda_{o(5.8\text{-GHz})} = 0.0517$ m) [8]. Note that D_p denotes the depth at which the power density of the microwaves is reduced to 1/e of its initial value, and depends strongly on temperature and on the frequency of the microwave radiation. The absorbing media becomes more transparent to microwaves at higher temperatures. Thus, the penetration depth of the microwaves is an important factor in microwave heating (see below).

Microwave Frequency Effects

At present, the most commonly used microwave frequency in organic syntheses is the 2.45-GHz frequency, also commonly used in microwave domestic ovens. The efficiency of microwave dielectric loss heating is governed by the frequency of the radiation and by the electric field strength (eqn. 1, second term). Hence, the frequency effect in microwave-assisted chemistry cannot be disregarded. The microwave frequency for industrial communications equipment is typically provided by the International Telecommunication Union (ITU) in the ISM band (Industrial Science Medical). This frequency is limited to chemical industrial use. The following microwave frequencies 2.45 ± 0.25 GHz, 5.80 ± 0.25 GHz and 24.125 ± 0.125 GHz are permitted in many countries. Variations in dielectric loss factors with changes in temperature for both the 2.45-GHz and 5.8-GHz MW frequencies have been examined in water using an Agilent Technologies HP-85070B Network Analyzer. Results are illustrated in Fig. (**1**) [9], which show the dielectric loss factors decreasing (ca. 72% for the 2.45-GHz and ca. 60% for the 5.80-GHz MW frequencies) with increasing temperature of the media from 22 ºC to 99 ºC. The penetration depth of the 2.45-GHz microwaves increased 2.7-fold from ca. 1.8 cm to 4.8 cm for a temperature rise from 22 ºC to 99 ºC, whereas penetration was rather shallow for the 5.80-GHz microwaves (from 0.34 cm to 0.62 cm; i.e. a 1.8-fold increase). Clearly, the frequency is an important factor that impacts on the efficiency of heating by microwaves. As such, this brief review examines the microwave frequency effect(s) on a variety of solvents, both polar and non-polar, as well as the effect(s) on three organic reactions taken from our recent studies.

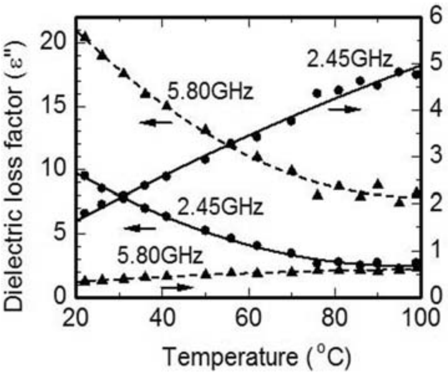


Fig. (1). Temperature profiles of the changes in the dielectric loss factor (ε'') and the penetration depth (cm) of the 2.45-GHz and 5.80-GHz microwaves into water. Taken from ref. [9]; Copyright 2009 by Elsevier B.V.

EXPERIMENTAL SETUP

5.80-GHz Microwave Apparatus for Chemical Reactions

Microwave frequency effects were examined using a newly fabricated 5.80-GHz microwave apparatus for organic syntheses [10]. The 5.80-GHz microwave frequency generator was obtained from Panasonic (Model M5801; maximal power, 700 Watts). The high-pressure Pyrex glass cylindrical reactor {size: 160 mm (H) × 37 mm (i.d.); maximal pressure, 1 MPa; maximal volume, 150 mL} was introduced into the metal pipe connected to the waveguide (see Fig. **2**), and then irradiated with the generated 5.80-GHz microwaves in the single mode. The waveguide was Japan industrial standard WRJ-6 {inner size, 40 mm × 20 mm}. A pressure gauge and a release bulb were connected to the cover of the reactor. The temperature of the solution in the reactor was monitored by an optical fiber thermometer (FL-2000, Anritsu Meter Co. Ltd.) and a k-type sheathed thermocouple. The cylindrical reactor was sealed with either a Viton or a silicon O-ring and the cover was made of Teflon. The choice of the O-ring was based on the nature of the solvent used. The system could be used either with a reflux condenser (Fig. **2a**) as a batch system (Fig. **2b**), or as a flow system using an appropriate pump. The frequency effect(s) of the 5.80-GHz microwaves was compared with the effect(s) from the more commonly used 2.45-GHz microwaves. In the latter case, the single mode microwaves also irradiated the reactor through the waveguide whose dimensions were: inner size, 96 mm x 27 mm {Japan industrial standard WST-AD}. The incident and reflected power levels of the microwaves were measured by the power monitor located in front of the reactor. Details of the 2.45-GHz microwave system were reported earlier [11]. The cross sections of the waveguides for the 5.80-GHz and 2.45-GHz microwaves are 80 mm^2 and 1592

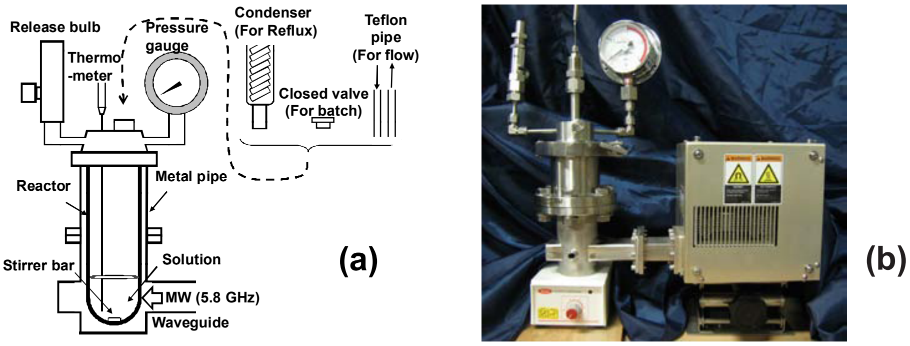


Fig. (2). (**a**) Schematic illustration of the reflux system and the 5.8-GHz microwave apparatus operated in the single mode; (**b**) actual photograph of the batch system using the 5.8-GHz microwaves to drive a chemical reaction system. Reproduced from ref. [10]; Copyright 2008 by the American Chemical Society.

mm^2, respectively; the difference in electric field density was ca. 32-fold under otherwise identical applied microwave power. Therefore, the 5.80-GHz microwaves concentrate microwave irradiation to the sample, resulting in increased efficient heating. On the other hand, the 2.45-GHz microwaves can irradiate a larger area of the sample.

RESULTS AND DISCUSSION

Frequency Effect(s) on Solvents

The behavior of frequency effect (5.8-GHz and 2.45-GHz) was examined for ion-exchanged water and for some 22 different types of common organic solvents. They are listed in Table **1**. Experiments involved a 30-mL sample of the solvents that was introduced into the Pyrex glass cylindrical reactor. All organic solvents were used after having been purified by distillation. The same distilled solvents were used for experiments with the 5.80-GHz and 2.45-GHz microwaves at a power of 30 Watts under continuous irradiation and under non-stirring conditions. The increase in temperature caused by the microwave radiation was monitored at 5 s intervals with a fiber-optic thermometer and a thermocouple. The rates of temperature increase for water and for some 22 common organic solvents under non-stirring conditions when subjected to 5.80-GHz and 2.45-GHz microwave radiation are reported in Table **1**. Note that for dichloromethane and diethyl ether the data were obtained for 1 min of microwave irradiation.

Results show that the rate of temperature rise with the 5.80-GHz microwaves is more than 2-fold faster than for the 2.45-Hz microwaves for *n*-pentane, diethyl ether, dichloromethane, cyclohexane, benzene, triethylamine, hexane, THF, ethyl acetate and

Table 1. Rates of Temperature Rise for Less Than 3 Min of MW Irradiation, and Ratio of Rates for Water and 22 Common Organic Solvents Under Non-Stirring Conditions[a]

Solvents	**Rates of temperature rise in < 3 min (°C min^{-1})**		**Ratio of rates (5.80 GHz/ 2.45-GHz)**	**Nature of solvents** [c]
	5.80-GHz	**2.45-GHz**		
n-pentane	2.65	0.10	26.5	Non-polar
diethyl ether [b]	18.1	1.81	10.0	
dichloromethane [b]	24.2	3.28	7.37	
cyclohexane	3.53	0.52	6.80	
benzene	3.29	0.54	6.13	
triethylamine	11.4	1.92	5.95	
hexane	2.66	0.47	5.63	
THF	15.8	4.76	3.32	
ethyl acetate	18.9	7.66	2.47	
xylene	8.91	4.18	2.13	
toluene	6.96	4.35	1.60	
pyridine	17.2	11.4	1.52	Polar
acetic acid	16.8	11.3	1.49	
acetone	16.1	11.6	1.39	
water	8.46	7.15	1.18	
acetic anhydride	14.6	12.6	1.16	
DMSO	14.1	12. 5	1.13	
2-propanol	15.8	15.5	1.02	
DMF	14.8	15.1	0.98	
methanol	13.8	14.8	0.93	
ethylene glycol	11.3	12.2	0.92	
ethanol	15.2	16.5	0.92	
1-propanol	14.5	16.3	0.89	

[a] Reproduced from ref. [10]. [b] For diethyl ether and dichloromethane rate determined after 1 min of microwave irradiation. [c] From ref. [12]

xylene. In particular, for diethyl ether the rate of microwave heating was 10-fold faster, whereas for *n*-pentane it was nearly 27-fold faster when utilizing the 5.80-GHz microwaves relative to the 2.45-GHz radiation. By contrast, for 1-propanol, ethanol, ethylene glycol, and methanol, together with DMF, the rate of increase was somewhat greater for the 2.45-GHz microwaves. It is noteworthy that for the polar solvents, differences in the rates of heating by a change in the frequency of the microwave radiation (5.8-GHz versus 2.45-GHz) tended to be rather small (see 4th column of Table **1**). Even for such a polar molecule as water the rate was 18% faster under 5.80-GHz microwave irradiation.

Superheating is known as one of the characteristics of microwave heating [13]. Superheating refers to the difference between the boiling points reached by microwave heating and conventional heating. In the case of the 5.80-GHz microwave heating, the time to reach the boiling point in less than 3 min was seen for ethyl acetate, THF, acetone, dichloromethane, and diethyl ether. By contrast, heating of ethyl acetate and dichloromethane by the 2.45-GHz microwave radiation required more than 30 min to reach the corresponding boiling points. Superheating was observed for all organic solvents exposed to the 5.80-GHz microwave radiation and for most polar solvents subjected to 2.45-GHz microwaves. Solvents that displayed a difference greater than 20ºC between the actual temperatures and the boiling points were ethyl acetate, acetic acid, 1-propanol, DMF, THF, acetone, and dichloromethane. These results indicate that superheating effects are most notable for the 5.80-GHz microwaves.

The most important characteristics of the 23 solvents examined with the two microwave frequencies at ambient temperature are the penetration depth of the microwaves into the solution bulk and the dielectric parameters of these solvents, namely the dielectric constant (ε'), the dissipation factors (sometime also referred to as the dissipation loss tangent, tan δ) and the dielectric loss factors (ε''). Note that the dielectric loss factors include the leak currents. The trend in the variations of the dielectric loss factors with microwave frequency paralleled the trend in the temperature rise. Changes in dielectric loss against frequency from 0.1 to 5.80-GHz are depicted in Fig. (**3**). For non-polar solvents the dielectric loss increases with increase in the frequency of the microwaves as shown in Fig. (**3a**). On the other hand, the trends for polar solvents showed somewhat large variations (Fig. **3b**). Comparison of the ratios of rates of temperature increase of Table **1** indicates that DMSO, water, and DMF displayed the largest dielectric losses (ε'') at the 5.80-GHz frequency, whereas the dielectric loss factors for methanol, ethylene glycol, and ethanol were greater at 2.45-GHz than at 5.80-GHz. Accordingly, the smaller ratios of rates for the alcohols and ethylene glycol at 2.45-GHz were induced by the higher dielectric loss factors at this frequency. The variations of the penetration depths of the 2.45-GHz and 5.8-GHz microwaves into each solvent, estimated using the dielectric parameters, showed a clear correlation with differences in the rates of temperature rise.

In summary, the effect(s) of the microwave frequency on organic solvents was somewhat remarkable, especially for non-polar solvents. Such effect(s) impacts strongly on the penetration depth of the microwaves.

Demonstrated Frequency Effect in Organic Synthesis

Application to a Diels-Alder Reaction

The Diels-Alder synthesis of 3,6-diphenyl-4-*n*-butylpyridazine from diphenyltetrazine and 1-hexyne reagents in non-polar solvent (ethyl acetate and xylene) is illustrated in eqn. **3**, whereas the chemical yields under various experimental heating conditions after silica-gel treatment of the resulting product(s) are listed in Table **2** [14].

The yield of 3,6-diphenyl-4-*n*-butylpyridazine under irradiation with 5.80-GHz microwaves was 8-12% in ethyl acetate solvent; a similar yield (10-12%) was obtained with xylene as the solvent. This was not unexpected for the synthesis of this pyridazine since irradiation in these two solvents with 2.45-GHz microwaves was rather inefficient when looking at the temperature increase with irradiation time. Further irradiation also led to no reaction. By contrast, heating the reaction solution in xylene with an oil bath led to a 2–5% yield of 3,6-diphenyl-4-*n*-butylpyridazine; note that the reactor had been placed in the oil bath preheated at 80 ºC because the initial rate of temperature rise was somewhat slow.

In xylene solvent, the reaction temperature upon oil-bath heating was higher than that observed when heating with 5.80-GHz microwaves. The temperature-time profiles of the reacting sub-

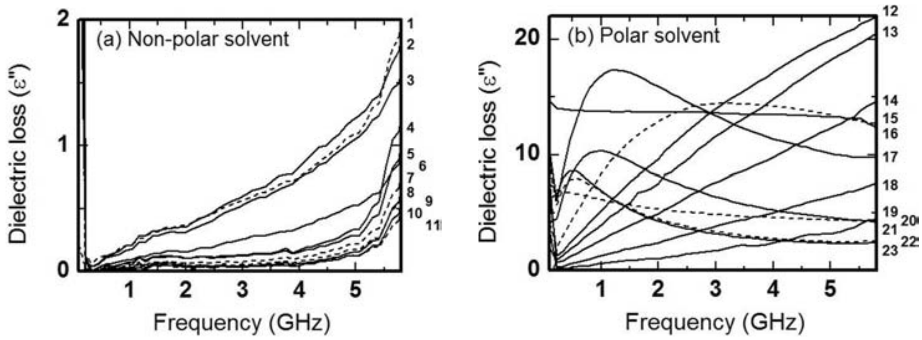
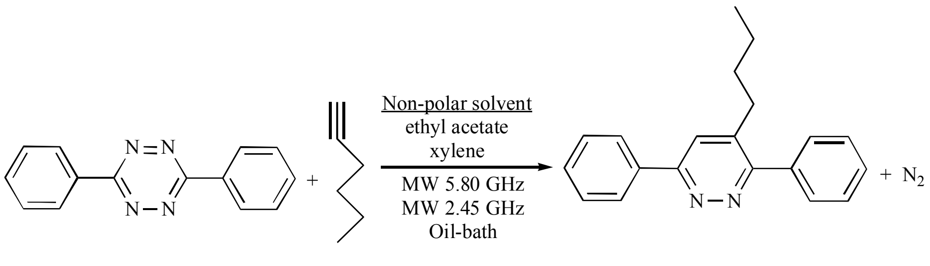


Fig. (3). Temporal changes in dielectric loss factors (ε'') for frequencies 0.1 GHz to 5.80-GHz. (1) ethyl acetate, (2) dichloromethane, (3) THF, (4) triethylamine, (5) diethyl ether, (6) xylene, (7) toluene, (8) cyclohexane, (9) n-pentane, (10) benzene, (11) hexane, (12) DMSO, (13) water, (14) DMF, (15) pyridine, (16) methanol, (17) ethylene glycol, (18) acetic anhydride, (19) ethanol, (20) acetone, (21) acetic acid, (22) 2-propanol, and (23) 1-propanol.



(3)

Table 2. Yields of 3,6-diphenyl-4-n-butylpyridazine (DBP) Under Various Experimental Conditions

Heating Method	Solvent	Temperature (°C)	% Yield after	
			60 min	90 min
5.80-GHz	ethyl acetate	81	8	12
	xylene	122	10	12
2.45-GHz	ethyl acetate	43	0	0
	xylene	33	0	0
Oil bath	ethyl acetate	76	0	<1
	xylene	135	~2	5

strates in an open reactor system are displayed in Fig. (**4**). The temperature of naked 3,6-diphenyl-1,2,4,5-tetrazine (tetrazine) and 1-hexyne increased rapidly under irradiation with 5.80-GHz microwaves. The temperature of tetrazine reached 120 ºC when microwave irradiated for 90 sec. By contrast, the temperature increases of the solvents were somewhat attenuated for the 2.45-GHz microwaves. The rapid progress of the synthesis of 3,6-diphenyl-4-*n*-butylpyridazine by the 5.80-GHz microwave radiation must be due to the relatively rapid heating of the reacting substrates and the non-polar solvent.

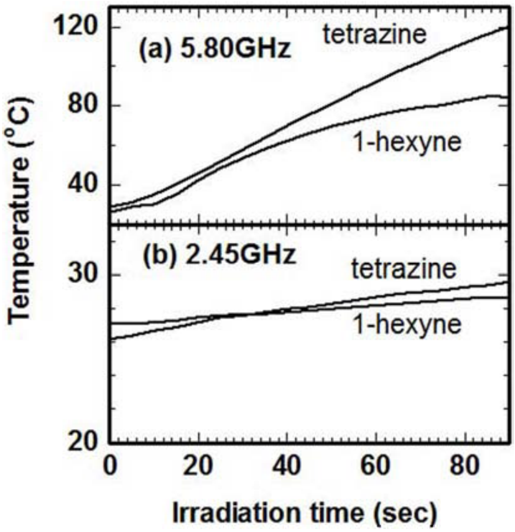


Fig. (4). Temperature-time profiles of naked 3,6-diphenyl-1,2,4,5-tetrazine and 1-hexyne when subjected to 5.80-GHz and 2.45-GHz microwave irradiation.

Synthesis of a Room-Temperature Ionic Liquid (RTIL)

Both 5.80-GHz and 2.45-GHz microwaves were used to carry out the novel one-pot solvent-free synthesis of the RTIL 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4) in a reactor containing the three required reagents, namely 1-methylimidazole, sodium tetrafluoroborate and 1-chlorobutane (eqn. **4**) [15]. The synthesis yields of this ionic liquid under the various experimental conditions used are listed in Table **3**.

The chemical yields of [bmim]BF_4 (84-87% after a 30 and 60 min) from 5.80-GHz microwave heating were 3- to 4-fold greater than the yields obtained with the 2.45-GHz microwaves at two different times (ca. 28-29%), even though the temperatures were lower at the higher microwave frequency. The temperature-time profiles of the mixture of reacting substrates in the synthesis of [bmim]BF_4 under both microwave frequency radiation were quite dissimilar. After 10 min into the process, heating with the 5.80-GHz microwaves was constantly lower than with the 2.45-GHz microwaves. The temperature increase was not monotonic at the latter frequency. In fact, for the synthesis of [bmim]BF_4 by the batch process, unusual temperature increases were observed with the 2.45-GHz radiation after 3, 10 and 20 min. This irregular increase was likely due to the formation of the [bmim]BF_4 ionic liquid. That is, the heating efficiency with the 2.45-GHz microwaves changed because the composition of the reacting mixture changed by formation of [bmim]BF_4. Contrary to the latter, the temperature-time profiles observed for the 5.80-GHz microwave radiation was somewhat noisy beyond 20 min of irradiation, but otherwise of no consequence with regard to the smooth formation of the product. The temperature-time profiles of the individual reacting substrates 1-chlorobutane (CB), 1-methylimidazole (MI), and the solid $NaBF_4$ in the batch mode showed significant differences on irradiation with the 2.45-GHz microwaves (CB: 84 ºC, MI: 147 ºC, and $NaBF_4$: 56 ºC), respectively, after a 10-min irradiation period. Differences in the temperature-time profiles of the reacting materials subjected to

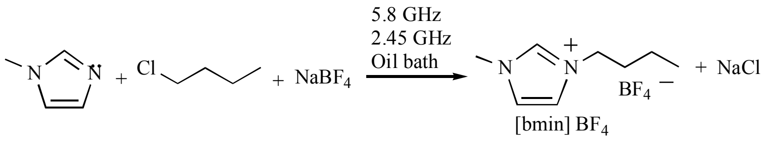


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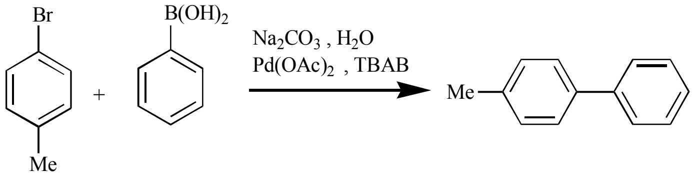
Table 3. Chemical Yields of [bmim]BF_4 from the Solvent-Free One-Pot Synthesis Under Various Experimental Conditions

Microwave Frequency	Time (min)	Temperature (°C)	% Yield
5.80 GHz	30	155	87
	60	169	84
2.45 GHz	30	204	28
	60	219	29



(5)

the 5.80-GHz microwave radiation were smaller (CB: 105 ºC, MI: 124 ºC and $NaBF_4$: 143 ºC), respectively, after 10 min) than with the 2.45-GHz microwaves. Closer examination of the results also revealed that the temperature rise in the synthesis of [bmim]BF_4 with the 2.45-GHz microwaves was more effective than with the 5.80-GHz microwaves.

The Suzuki-Miyaura Coupling Reaction in a Polar Solvent

The microwave-enhanced synthesis of 4-methylbiphenyl from 4-bromotoluene and phenylboronic acid in the presence of a $Pd(CH_3CO_2)_2$ catalyst in water was examined under both the 5.80-GHz and 2.45-GHz microwave radiation and by conventional heating using an oil bath (eqn. **5**). The relevant temperature–time profiles are reported in Fig. (**5**). The power levels for the 5.80-GHz microwaves (61 Watts) and for the 2.45-GHz microwaves (100 Watts) were chosen in such a way that the rates of temperature increase coincided and that the temperature reached after 260 s was the same (92 ºC). On the other hand, the temperature of the solution was 100 ºC when using 5.80-GHz microwave at an applied power of 100 Watts. In the case of oil-bath heating, the reactor was placed into the pre-heated oil bath at 190 ºC, and then the temperature rise of the solution determined.

Chemical yields of 4-methylbiphenyl obtained under various experimental conditions are listed in Table **4**. The yields of the biphenyl product were substantive after only 10 min of microwave irradiation: 84% with the 5.80-GHz microwaves (100 Watts), 75% with the 5.80-GHz microwaves but at a power of 61 Watts, 73% with the 2.45-GHz microwaves (100 Watts), and 61% when the reaction was heated conventionally with an oil bath also after 10 min. That the yield by the 5.80-GHz microwaves is somewhat higher than for the 2.45-GHz microwaves under identical applied power levels of 100 Watts was likely due to the difference in the reaction temperatures. However, no difference in chemical yields was observed when the reaction occurred at the same temperature but heating provided at two different microwave frequencies. Note that most of the microwaves were used to heat the solvent (water).

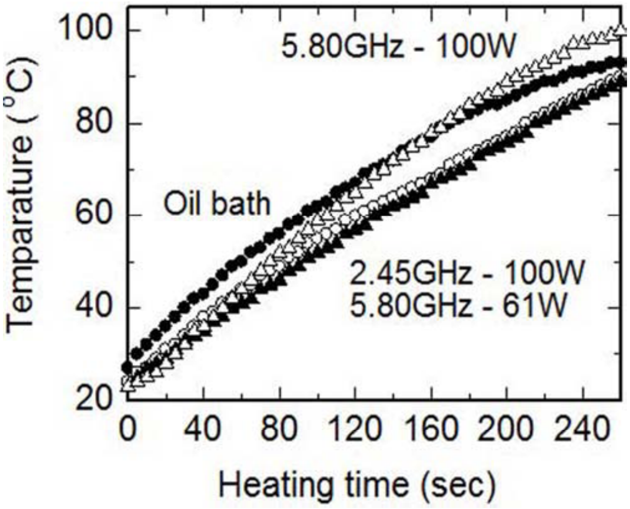


Fig. (5). Temperature-time profiles in the synthesis of 4-methylbiphenyl on irradiation with the 5.8-GHz microwaves (power, 61 Watts and 100 Watts) and with the 2.45-GHz microwaves (100 Watts) and with oil bath heating.

Table 4. Chemical Yields of 4-methylbiphenyl with Water Solvent Under Various Heating Conditions

	Time (min)	% yield
5.80-GHz (100 Watt)	5	37
	10	84
5.80-GHz (61 Watt)	5	31
	10	75
2.45-GHz (100 Watt)	5	30
	10	73
Oil-bath	5	20
	10	61

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