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Use of Microwave Heating in Coal Research and in Materials Synthesis

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1. Introduction

In this chapter, the use of microwave heating in addressing some problems in coal research and in the synthesis of inorganic materials are reviewed. The review covers the underlying theory of microwave heating and how it has been used in specific cases. This review is divided into three sections: Section 2 dealing with the fundamental theory behind microwave heating and section 3 and 4 dealing with its use in coal research and in the synthesis of materials respectively. References to selected papers are also given. However this list of references is not exhaustive and readers are asked to consult these references for earlier studies in specific areas.

2. Fundamentals of microwave heating

When a time varying electric field $E = E_o\exp(i\omega t)$ is applied to a liquid or solid, polarization develops which in general can come from three sources viz. dipolar, ionic and electronic (Kittel, 1996). The dipolar contribution is present in polar materials viz. materials with permanent dipoles such as water as these dipoles re-orient in the direction of the applied field E. The relaxation time τ associated with this reorientation is in picoseconds (ps) and therefore this phenomena is observable when frequency $f = (\omega/2\pi)$ is in the microwave range (f = 0.1 GHz to 100 GHz). The ionic contribution to the polarization results from the various normal modes of vibration of the atoms relative to each other. These frequencies in the IR range are from 300 GHz to 100 THz. Finally, the electronic contribution resulting from the relative displacements of the nucleus with positive charge and electron shell of the atoms occurs in the ultraviolet (UV) region of the frequencies. Thus microwave heating as shown below is most significant in materials having permanent dipoles such as water.

A number of experimental studies (Kaatze, 1989; Merabat & Bose, 1988) have shown that dielectric relaxation in water is adequately described by the Debye relation (Debye, 1929)

$$\varepsilon(\omega) = \varepsilon(\infty) + [\varepsilon(0) - \varepsilon(\infty)]/(1 + i\omega\tau) \quad (1)$$

where $\varepsilon(0)$ and $\varepsilon(\infty)$ are the dielectric constant in the limit $\omega\rightarrow 0$ and $\omega\rightarrow\infty$ respectively. For water, $\varepsilon(\infty)$ includes the polarization effects at the infrared and optical-uv frequencies. Separating the in phase and the out of phase components of $\varepsilon = \varepsilon' - i\varepsilon''$ in Eq.(1) leads to

$$\varepsilon' = \varepsilon(\infty) + [\varepsilon(0) - \varepsilon(\infty)]/(1 + \omega^2\tau^2) \quad (2)$$

and

$$\varepsilon'' = \omega\tau[\varepsilon(0) - \varepsilon(\infty)]/(1 + \omega^2\tau^2) \quad (3)$$

Eliminating $\varepsilon(\infty)$ through substitutions in Eq. (2) and (3) leads to the following relation between $\varepsilon'$and ε'':

$$\varepsilon' = \varepsilon(0) - \varepsilon''\cdot\omega\cdot\tau \quad (4)$$

Once experimental data for ε' and ε'' are available for different ω, a plot of ε' vs. $\varepsilon''\cdot\omega$ allows one to determine the relaxation time τ. Such an analysis for water in the frequency range of 1 GHz to 60 GHz and in the temperature range of 0˚C and 60˚C have shown (Kaatze, 1989) that at T = 25˚C, τ = 8.27 ps, $\varepsilon(0)$ = 78.36 and $\varepsilon(\infty)$ = 5.2 and that with increase of temperature, $\varepsilon(0)$, $\varepsilon(\infty)$ and τ decrease rapidly so that at 60˚C, $\varepsilon(0)$ = 66.7, $\varepsilon(\infty)$ = 4.2 and τ = 4.0 ps.

Microwave heating involves absorption of electrical power by water from the microwaves. The power absorbed P can be shown to be given by (Agmon, 1996)

$$P = 2\pi |E_o|^2 \varepsilon_o f\cdot\varepsilon'', \quad w/m^3 \quad (5)$$

where $\varepsilon_0 = 8.854 \times 10^{-12}$ A s/V m, E_o is the amplitude of the electric field and $\omega = 2\pi f$. The important point to note is that P is proportional to $f\cdot\varepsilon''$so that P depends upon the frequency f of the microwaves and $\varepsilon''$of the medium (water). From Eq. (3) it can be easily shown that ε'' peaks at $\omega\tau = 1$.

In order to provide visualization of the frequency variation of the quantities ε', ε'' and $f\cdot\varepsilon''$, the latter representing the power absorbed P, we have used the data for water given in a tabular form (Kaatze, 1989). The simulated plots of the frequency dependence of ε', ε'' and $f\cdot\varepsilon''$ for the data valid at 25˚C are shown as lines in Fig. 1 where the circles are experimental values (Kaatze, 1989). The data up to 60 GHz fit quite well with the Debye relaxation with a single τ. Later studies have suggested that two relaxation times are necessary to explain the data at frequencies higher than 100 GHz (Barthel et al., 1990; Agmon, 1996).

An interesting feature of Fig. 1 is the frequency dependence of $f\cdot\varepsilon''$ representing power absorbed. This plot, usually not avaliable in literature, shows that power absorbed saturates only near 100 GHz. At f = 2.45 GHz, the conventional frequency used in microwave ovens, domestic and industrial, the absorbed power is only about 1.6% of the power absorbed at 100 GHz. However, the wavelengths of microwaves λ = 0.3 cm at 100 GHz compared to λ = 12.2 cm at 2.45 GHz, thus reducing the size of the microwave ovens to impractical size of only about 0.3 cm at the higher frequency. At 17 GHz where ε'' has the maximum value of 36, λ = 1.76 cm and power absorbed is nearly 50% of the maximum value at 100 GHz.

As water heats up, τ, $\varepsilon(0)$ and $\varepsilon(\infty)$ decrease (Kaatze, 1989) affecting the peak positions for $\varepsilon''$and $f\cdot\varepsilon''$. In Fig. 2 simulated frequency dependence of ε', ε'' and $f\cdot\varepsilon''$ is compared at two temperatures viz. 25˚C and 60˚C using the magnitudes of $\varepsilon(0)$, $\varepsilon(\infty)$ and τ (Kaatze, 1989). The important point to note is that the power absorbed at a fixed frequency below about 30 GHz decreases as the temperature of water increases.

The frequency derivative of power absorbed by water given by $\partial(f\cdot\varepsilon'')/\partial f$, is plotted in Fig. 3. The maximum slope occurs at 11 GHz for 25˚C and the position of this maximum changes to 23 GHz at 60˚C. Analytically, using relation (3), it can be shown that $\partial(f\cdot\varepsilon'')/\partial f$ is maximum at $f = 1/\pi\tau\sqrt{12}$.

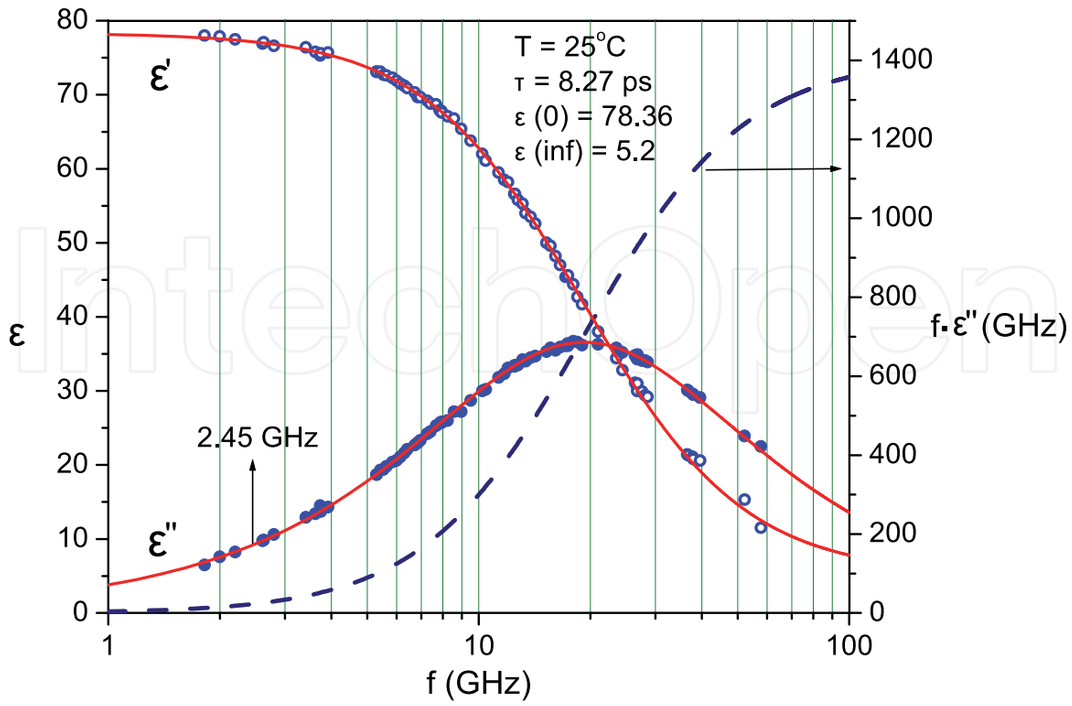

Fig. 1. The simulated plots of the frequency dependence of ε′, ε″ and f·ε″ for water using the data (circles) at 25˚C from Kaatze (1989).

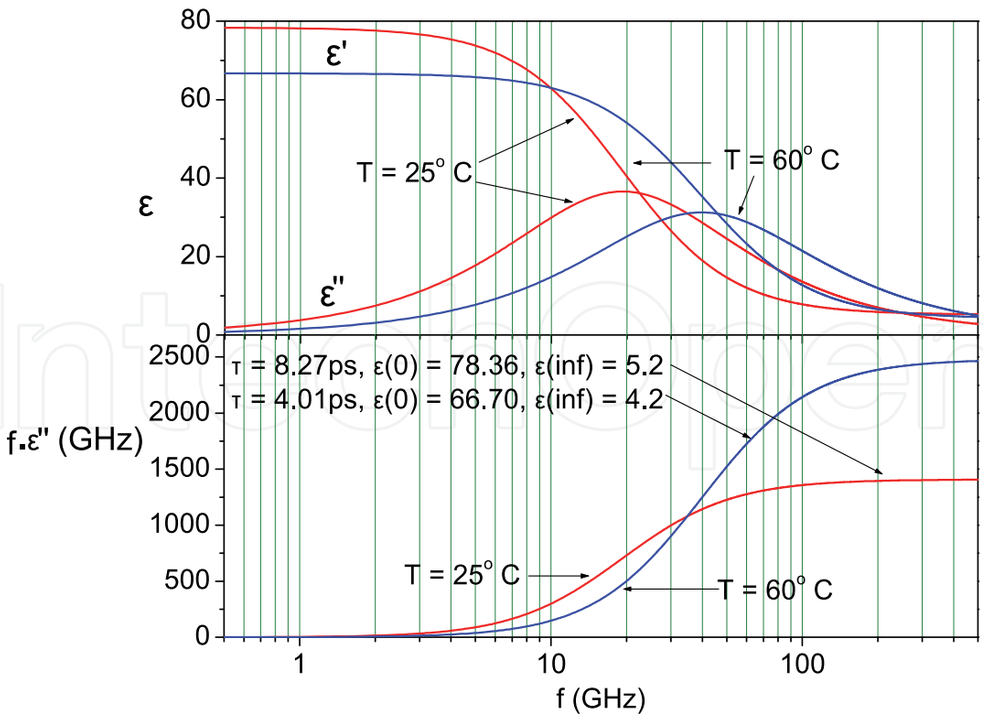

Fig. 2. Simulated frequency dependence of ε′, ε″ and f·ε″ is compared for water at two temperatures viz. 25˚C and 60˚C using the magnitudes of ε(0), ε(∞) and τ

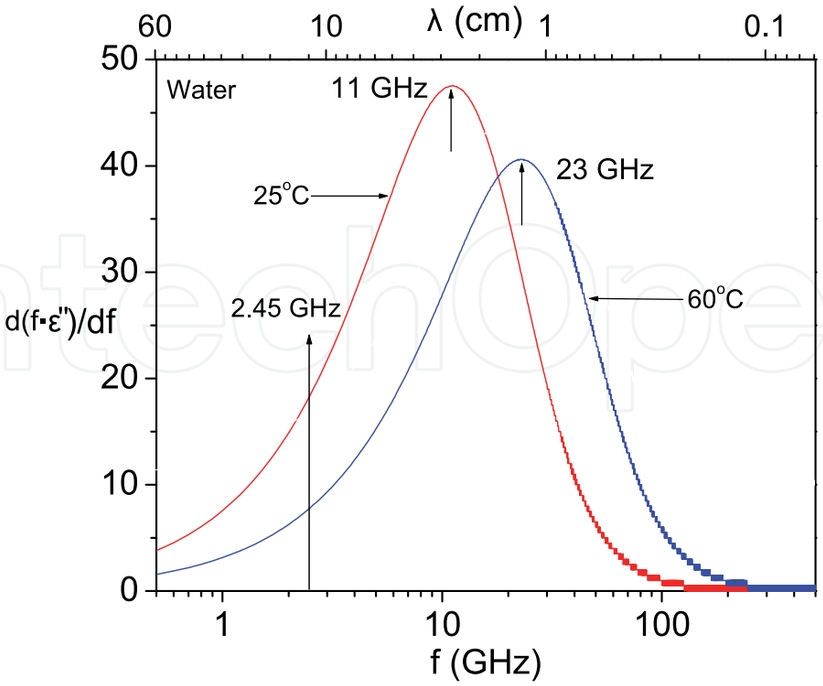

Fig. 3. Derivatives of power absorbed from Fig. 2 vs. frequency and wavelength at 25˚C and 60˚C.

3. Microwave heating in coal research

3.1 Microwave properties of coals

In the recent paper, (Marland et al., 2001) have reported the measurements of ε'and ε" at 60˚C for over a dozen coals and for the ash components normally found in most coals. In these studies, data were reported at f = 0.615 GHz, 1.413 GHz and 2.216 GHz for the coals and the minerals pyrite, quartz, dolomite, kaolin, mica and calcite. Although there is some variations in the magnitudes of ε' and ε" from coal to coal, the range of values were between 1.6 to 3.5 for ε' and 0.07 to 0.31 for ε"at f = 2.216 GHz. The frequency dependence among the three frequencies was found to be relatively weak. Among the minerals the highest magnitude was found to be for pyrite with ε'= 7.07 and ε"= 1.06 at 2.216 GHz and at 60˚C. For the other minerals, ε' ~ 2 to 3 and ε" ~ 0.05-0.06. Moisture levels in these coals varied from a low of 1% to a high of 13%. From Fig. 1 ε" ≃ 10 for water at 2.45 GHz. Since power P absorbed is proportional to f•ε" (Eq. 1), the highest power absorbed will be by moisture, followed by pyrite. Thus the largest effect of microwave heating of coal is the removal of moisture and heating of pyrite FeS_2.

Pyrite FeS_2 when heated above 250˚C is known to convert to pyrrhotites $Fe_{1-x}S$ (x = 0 - 0.125) whose mangetization at room temperature is ferrimagnetic like, with the magnitude depending on the x value (Jagadeesh & Seehra, 1981). Whereas FeS_2 is only paramagnetic with feeble magnetization (Burgardt & Seehra, 1977) pyrrhotites $Fe_{1-x}S$ are strongly magnetic (Jagadeesh & Seehra, 1981). Therefore conversion of pyrites to pyrrhotites after microwave heating make them amenable to separation by magnetic methods. Thus microwave heating of coals at an appropriate frequency followed by magnetic separation of the impurities can lead to both dewatering and desulphurization, since a major source of sulphur in coals is

pyrites. A combination of these techniques is likely being used in the recently developed and marketed technology by CoalTek (http://www.coaltek.com/).

3.2 Measuring moisture content by microwaves

Another area in which microwaves have been used in coal research is the on-line measurements of moisture in coals employing the frequencies of 2.45 GHz (Cutmore et al., 2000) and 1 GHz (Ponte et al., 1996). In these techniques, microwave horns are used to transmit and receive microwaves through the coal sample. The use of the lower microwave frequencies and hence corresponding larger wavelengths of λ = 12.2 cm (30 cm) for 2.45 GHz (1 GHz) allows the use of larger samples encountered at coal power plants. As microwaves pass through a coal sample, large ε' of water produces a frequency and phase shift of the microwaves and ε'' results in the attenuation of the microwave signal. Both phase shift and attenuation are measured by microwave network analyzer. However, experiments show that phase shift provides a better linear correlation with the moisture content (Ponte et al., 1996; Cutmore et al., 2000). Since attenuation is due to absorption of microwaves primarily by moisture present, this technique can also result in sufficient dewatering of the coals. This topic of dewatering or drying of coals by using microwave is discussed next.

3.3 Microwave dewatering of coals

Dewatering or drying of coals is an important consideration in coal industry since removal of moisture reduces the transportation costs and improves the BTU value of the coals. The use of microwaves for dewatering of coals has been discussed in a number of earlier studies (Lindroth, 1985; Chatterji & Mishra, 1991; Perkin, 1980; Standish el al., 1988; Marland el al., 2000; Lester & Kingman, 2004) and in a more recent study from author's laboratory (Seehra et al., 2007). In microwave dewatering of coals, the basic mechanism for power absorption by water discussed in Section 2 is operative.

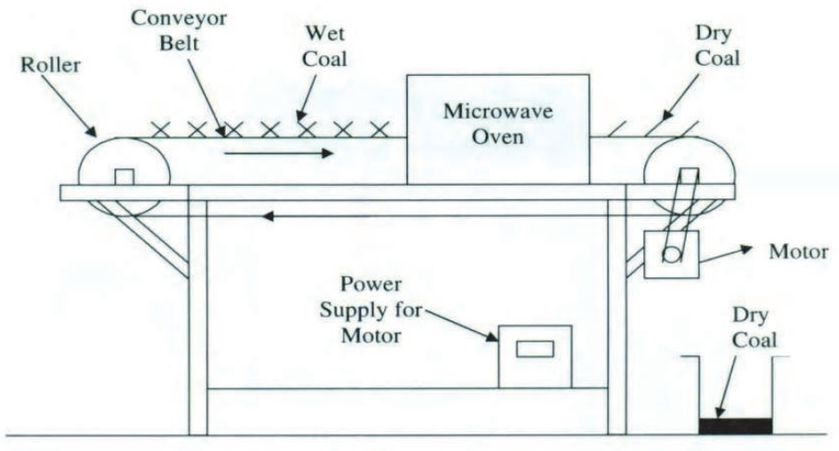

Fig. 4. Block diagram of the bench-scale microwave dewatering unit (reproduced from the paper by Seehra et al., Fuel 2007).

In our studies of microwave dewatering (Seehra et al., 2007), the focus was on dewatering fine coal slurries containing nearly 50% moisture. A domestic microwave oven operating at 2.45 GHz was modified, Fig. 4 and Fig. 5, so that a conveyer belt could be used to

continously feed wet coal into the oven and dried coal collected at the other end. The whole apparatus was shielded with a microwave shield. This bench scale unit was designed to simulate a practical situation at a coal plant. The website of CoalTek shows such a conveyer belt approach and perhaps the only way to achieve large scale continous operation.

Fig. 5. (Left) Picture of the bench-scale microwave dewatering units with microwave shield in place. (Right) Close-up of the unit without the microwave shield showing the conveyor belt for feeding wet coal into the microwave oven (reproduced from the paper by Seehra et al., Fuel 2007).

In our experiment using a 0.8 kW domestic oven, the amount of moisture removed depended strongly on the belt speed; The slower belt speed removed more moisture but consumed more energy. This data on the relation between belt speed, electrical energy consumed and more moisture lost is shown in Fig. 6 (Seehra et al., 2007). Belt speed of

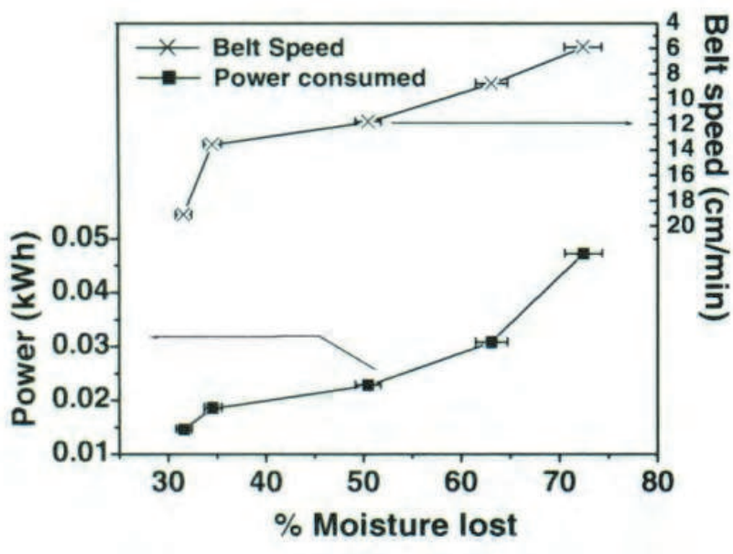

Fig. 6. Power consumed and belt speed (cm/min) vs. % moisture lost. The region of the least slope (13 cm/min) yields the optimum speed (reproduced from the paper by Seehra et al., Fuel 2007).

around 13 cm/min was found to be most energy efficient (~ 83%) for removal of water, the remaining power perhaps lost to surroundings and small heating of the minerals in coal and coal itself. In the experiments of Lindroth using higher power magnetrons (9.7 kW) operating at 2.45 GHz, the reported efficiency varied from 52% to 97% for drying of fine coals. A cost estimate of $ 3/ton to remove 10% moisture from coal was made based only on the amount of electrical power consumed (Seehra et al., 2007).

Dewatering efficiencies of microwave heating vis-à-vis thermal heating for the coal slurry samples are compared in Fig. 7 (Seehra et al, 2007). In this comparison, 20 gms of coal slurry sample was heated in a thermal oven set at 110˚C followed by taking the sample out after every 30 s for quick weighing and reinserting the sample in the oven in less than 5 s. Similar experiments were done using a domestic microwave oven. The results of weight loss vs. time in Fig. 7 show that complete dewatering of the coal slurry takes about 5 min with microwave heating compared to about an hour with thermal heating. Thus this experiment shows an order of magnitude reduction in time in microwave heating vis-à-vis thermal heating.

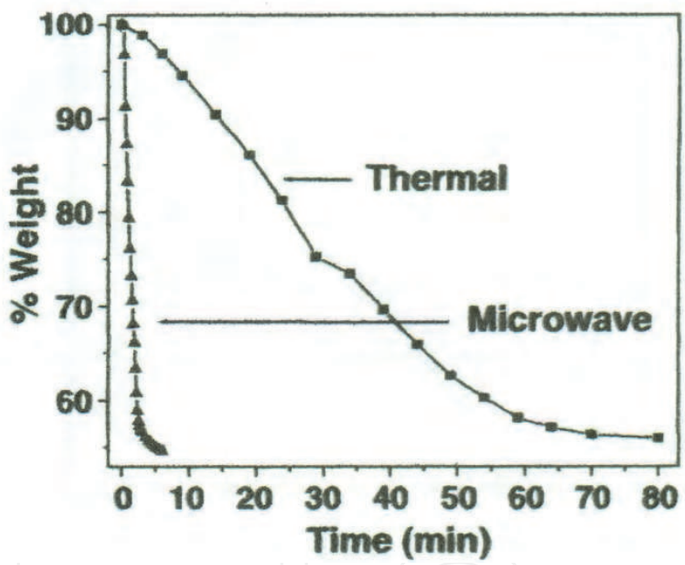

Fig. 7. Comparison of the efficiencies of thermal heating vis-a`-vis microwave heating using 20 g sample in each case (reproduced from the paper by Seehra et al., Fuel, 2007).

4. Use of microwaves in material synthesis

In section 2, the mechanism of microwave heating was described in terms of dielectric constants ε'and ε" with specific examples given for microwave heating of water. Same argument can be used for all polar materials with known ε'and ε". As an example, Barthel et al. (1990) have discussed the microwave heating using lower alcohols (methanol, ethanol, 1-propanol and 2-propanol).

In the chemical synthesis of a variety of materials, use of a conventional heating which has been a norm for a long time, is being replaced by microwave heating. In conventional heating, energy is first delivered to the surface of a material, followed by heat transfer to the material through conduction, convection and radiation. This results in non-uniform heating

producing poor crystallinity and often wider distribution of particle sizes. By contrast, in microwave heating, energy is transferred throughout the material via molecular interaction thus producing uniform heating without thermal gradients and stress. This often results in materials with finer microstructures and particles with narrow size distribution. The reader is referred to several recent papers (Barison et al., 2010; Wu et al., 2008; Prasad et al. 2002; Utchariyajit et al. 2010; Chang et al. 2009; Gopinath et al. 2002; Vincente et al 2010) where examples of these advantages are described in detail. Thus, compared to conventional thermal heating, microwave heating has the advantages of speed, energy efficiency, finer microstructures and more uniform particle size distribution in the synthesis of materials in line with the data shown in Fig. 7.

Microwave heating in combination with hydrothermal processing has shown positive effects on crystallinity of samples. Samples prepared by conventional hydrothermal treatment showed poor crystallinity in an amorphous phase, and they required higher temperatures and longer times than that of microwave hydrothermal treatment (Kim et al. 2001). Crystalline samples have been obtained without increase in particle size (Addamo et al. 2008). Other observations have shown the particles to have higher crystallinity indicating larger particle size (Vincente et al. 2009), and it avoids the post treatment such as annealing required after sol-gel synthesis (Hu et al. 2007). The treatment time under microwave heating has shown increase in the crystallite size due to Oswald mechanism of crystallite growth. Thus for applications which involve surface and phase characteristics, and the presence of porosity used in catalytic applications, microwave synthesis has been very successful (Wilson et al. 2006).

From a review done by Yanshou et al. in 2008, synthesis using microwave heating has been found to have the Si/Al ratio increased from 1.4 to 1.8 in zeolite membranes. It is clearly pointed out that the difference between microwave heating and conventional heating is due to different heating mechanisms (Li &Yang, 2008). Shape effects have also been noticed by Chen et al. 2009, where the concentration of solvents of diols have significantly affected the length of the Si-MFI crystals compared to the widths and thickness having a lesser effect. This effect could not be reproduced using a conventional synthesis method (Chen et al., 2009). In general, microwave hydrothermal treatment has proved to be more efficient than conventional hydrothermal treatment because of energy saving and time. For preparing microwave-assisted organic materials, readers are referred to the review by Lidström et al. (2001).

Experimentally, all materials placed inside a microwave oven during operation must be “microwave safe”, i.e.. they cannot be good electrical conductors, as is well known in the domestic use of microwave cooking. This is because the concentrated electric field associated with the microwaves directly flows through the good conductor resulting in electrical sparks and even fires. Ceramic and polymer containers which are good insulators and so “microwave safe” are now avaliable for use in material synthesis.

Historically, the earlier use of microwave heating in material synthesis involved the use of domestic microwave oven appropriately modified by each investigator. In recent years, microwave ovens specially designed for synthesis of materials are now avaliable in which temperatures and even pressures in an autoclave can be monitored in situ. The use of autoclaves inside the microwave ovens producing materials under hydrothermal conditions has greatly expanded the use of microwave heating. (e.g. see Chang et al. 2009 & Kim et al. 2001). Suppliers of such commercial units can be easily located by the readers in each country through internet search.

In the rest of this chapter on the use of microwave heating in the synthesis of materials, we have organized the available examples of such cases in a tabular form. The following table lists the name of the materials synthesized, conditions used in the synthesis and the corresponding reference covering the period from 2010 to 1998 with the most recent listed first. From this list, it is evident that the use of microwave heating in material synthesis is now becoming ubiquitous and it is sure to expand even more in the future.

SYSTEM	CONDITIONS	REFERENCES
$BaCe_{0.65}Zr_{0.20}Y_{0.15}O_{3-\delta}$	P = 350, 500 W	Barison S., Fabrizio M., Fasolin S., Montagner F. &Mortalò C., (2010). A microwave-assisted sol-gel Pechini method for the synthesis of $BaCe_{0.65}Zr_{0.20}Y_{0.15}O_{3-\delta}$powders. *Mater. Res. Bull.*, Vol. 45, pp. 1171-1176.
$Zn_4O(BDC_3)$	t = 30 min, P = 1 kW	Lu C., Liu J., Xiao K.& Harris A., (2010). Microwave enhanced synthesis of MOF-5 and its CO_2 capture ability at moderate temperatures across multiple capture and release cycles. *Chem. Eng. J.*, Vol. 156, pp. 465-470.
Capillary MFI-type Zeolite-Ceramic Membrane	T = 70 - 180 ˚C, P = 250 or 400 W, t = 10 - 150 min	Sebastian V., Mallada R., Coronasa J., Julbe A., Terpstra R.&Dirrix R., (2010). Microwave-assisted hydrothermal rapid synthesis of capillary MFI-type zeolite-ceramic membranes for pervaporation application.*J. Membrane Sci.*, Vol. 355, pp. 28-35.
Gold Nanoparticles	t = 30 sec, P = 800 W t = 9 min, P = 1 kW	Vargas-Hernandez C., Mariscal M., Esparza R. &Yacaman M., (2010). A synthesis route of gold particles with using a reducing agent.*Appl. Phys. Lett.* Vol. 96, pp. 213115-(1-3).
Silicalite (Si-MFI) Crystals	T = 180 ˚C, P = 400 W, t = 10 min	Chen X., Yan W., Cao X. &Xu R., (2010). Quantitative correlation between morphology of silicalite-1 crystals and dielectric constants of solvents. *Microporous andMesoporous Materials*, Vol. 131, pp. 45-50.
Saponite $M_x[Mg_6Al_xSi_{8-x}O_{20}(OH)_4]$ (M = Na, Li, NH_4)	T = 453 K, t = 6 hrs	Vicente I., Salagre P., Cesteros Y., Medina F.&Sueiras J., (2010). Microwave-assisted synthesis of saponite. *Appl. Clay Sci.*, Vol. 48, pp. 26-31.
$K_8Ln_3Si_{12}O_{32}NO_3 \cdot H_2O$ (Ln = Eu, Tb, Gd, Sm).	T = 363 K, t = 20 min	Wang X., Li J., Wang G., Han Y., Su T., Li Y., Yu J.&Xu R., (2010). Synthesis, characterization and properties of microporous lanthanide silicates:$K_8Ln_3Si_{12}O_{32}NO_3 \cdot H_2O$ (Ln = Eu, Tb, Gd, Sm). *Solid State Sciences*, Vol. 12, pp. 422-427.

Calcia Stabilized Zirconia Nanoparticles	T = 200 ˚C, t = 90 min	Rizzuti A., Corradi A., Leonelli C., Rosa R., Pielaszek R. &Lojkowski W., (2010). Microwave technique applied to the hydrothermal synthesis and sintering of calcia stabilized zirconia nanoparticles. *J. of Nanopart. Res.*, Vol. 12, pp. 327-335.
Double-walled Carbon Nanotubes	T = 200 ˚C, t = 20 min	Dunens O. M., MacKenzie K. J. & Harris A. T., (2010). Large-Scale Synthesis of Double-Walled Carbon Nanotubes in Fluidized Beds. *Ind. Eng. Chem.Res.*, Vol. 49, No. 9, pp. 4031-4035).
One-Dimensional Nano-Structured Silver Titanates	T = 200 ˚C, P = 300 W, t = 1 hrs	Li Q., Kako T. & Ye J., (2010). Strong adsorption and effective photocatalytic activities of one-dimensional nanostructured silver titanates.*Appl. Catalysis A: General*, Vol. 375, pp. 85-91.
Mesoporous Zeotype SAPO-5 (Silicoaluminophosphate)	T = 180 - 200 ˚C, t = 0.5 - 2 hrs	Utchariyajit K. &Wongkasemji S., (2010) Effect of synthesis parameters on mesoporous SAPO-5 with AFI-type formation via microwave radiation using alumatrane and silatrane precursors. *Microporous and Mesoporous Materials*, Vol. 135, pp. 116-123.
$Ru/La_{0.75}Sr_{0.25}Cr_{0.5}Mn_{0.5}O_{3-\delta}$	P = 750 W, t = 1 min	Barison S., Fabrizio M., Mortalò C., Antonucci P., Modafferi V.&Gerbasi R., (2010). Novel $Ru/La_{0.75}Sr_{0.25}Cr_{0.5}Mn_{0.5}O_{3-\delta}$ catalysts for propane reforming in IT-SOFCs. *SolidState Ionics*, Vol. 181, pp. 285-291.
Tungsten Oxide Nanorods	T = 180 ˚C, t = 20 min	Li Y., Su X., Jian J. & Wang J., (2010). Ethanol sensing properties of tungsten oxide nanorods prepared by microwave hydrothermal method. *Ceramics International*, Vol. 36, pp. 1917-1920.
Saponites with Mg^{2+}	T = 180 ˚C, P = 600 W, t = 1, 2, 3, 4, 8 or 16 hrs	Trujillano R., Rico E., Vicente M., Herrero M. & Rives V., (2010). Microwave radiation and mechanical grinding as new ways for preparation of saponite-like materials. *Appl. Clay Sci.*, Vol. 48, pp. 32-38.
Titanate Nanotubes	T = 403 K, P = 70, 400 or 700 W, t = 3 hrs	Chen Y., Lo S. &Kuo J., (2010). Pb(II) adsorption capacity and behavior of titanate nanotubes made by microwave hydrothermal method.*Colloids and Surfaces A:Physicochemical and Engineering Aspects*, Vol. 361, pp. 126-131.

Mg, Al-Layered Double Hydroxides	T = 125 ˚C, t = 10 - 180 min	Benito P., Herrero M., Labajos F. & Rives V., (2010). Effect of post-synthesis microwave hydrothermal treatment on the properties of layered double hydroxides and related materials. *Appl. Clay Sci.*, Vol. 48, pp. 218-227.
Y_2O_3 and Yb-Y_2O_3	T = 140, 170 ˚C, P = 150 - 300 W, t = 5 - 22 min, 2 hrs	Serantoni M., Mercadelli E., Costa A., Blosi M., Esposito L.&Sanson A., (2010). Microwave-assisted polyol synthesis of sub-micrometer Y_2O_3 and Yb-Y_2O_3 particles for laser source application. *Ceramics International*, Vol. 36, pp. 103-106.
Co-Zn-Al catalysts	T = 125 ˚C, t = 10 - 300 min	Benito P., Herrero M., Labajos F. M., Rives V., Royo C., Latorre N. &Monzon A., (2009). Production of carbon nanotubes from methane: Use of Co-Zn-Al catalystsprepared by microwave-assisted synthesis. *Chemical Engineering Journal*, Vol. 149, pp. 455-462.
Hectorite $M_x[Li_xMg_{6-x}Si_8O_{20}(OH)_4]$ (M = Na, Li, NH_4)	T = 393 K, t = 16 hrs	Vicente I., Salagre P., Cesteros Y., Guirado F., Medina F.&Sueiras J., (2009). Fast microwave synthesis of hectorite. *Appl. Clay Sci.*, Vol. 43, pp. 103-107.
Titania Nanowires	T = 210 ˚C, P = 350 W, t = 5 hrs	Chang M., Lin C., Deka J., Chang F. & Chung C., (2009). Nanomechanical characterization of microwave hydrothermally synthesized titania nanowires. *J. Phys. D:Appl. Phys.*, Vol. 42, pp. 145105.
Graphitic Carbon Particle Chains	T = 160 - 220 ˚C, t = 40 min	Harris A., Deshpande S. &Kefeng X., (2009). Synthesis of graphitic carbon particle chains at low temperatures undermicrowave irradiation. *Materials Lett.*,Vol. 63, pp. 1390-1392.
Silicalite (Si-MFI)	T = 80 ˚C, P = 250 W, t = 90 min T = 180 ˚C, P = 400 W, t = 60 min	Chen X., Yan W., Cao X., Yu J. &Xu R., (2009). Fabrication of silicalite-1 crystals with tunable aspect ratios by microwave assisted solvothermal synthesis. *Microporous and Mesoporous Materials*, Vol. 119, pp. 217-222.
$ZnWO_4$ Nanoparticles	T = 160 ˚C, t = 1, 3 hrs	Bi J., Wu L., Li Z., Ding Z., Wang X. & Fu X., (2009). A facile microwave solvothermal process to synthesize ZnWO4 nanoparticles. *J. Alloys and Compd.*, Vol. 480, pp. 684-688.
Multiferroic $BiFeO_3$	t = 30 min, P = 800 W	Prado-Gonjal J., Villafuerte-Castrejón M., Fuentes L. &Morán E., (2009). Microwave-hydrothermal synthesis of the multiferroic $BiFeO_3$. *Mater. Res. Bull.*, Vol. 44,pp. 1734-1737.

$NaYF_4:Yb^{3+},Tm^{3+}$	T = 180 ˚C, t = 4 hrs	Chen X., Wang W., Chen X., Bi J., Wu L., Li Z.&Fu X., (2009). Microwave hydrothermal synthesis and upconversion properties of $NaYF_4:Yb^{3+},Tm^{3+}$ with microtube morphology. *Mater. Lett.*,Vol. 63, pp. 1023-1026.
Carboxylate-Intercalated Layered Double Hydroxides	T = 85, 100, 125 ˚C, t = 10, 30, 180 min	Benito P., Labajos F., Mafra L., Rocha J. &Rives V., (2009). Carboxylate-intercalated layered double hydroxides aged under microwave-hydrothermal treatment. *J. Solid State Chem.*, Vol. 182, pp. 18-26.
Rapid Synthesis of Titania Nanowires	T = 210 ˚C, P = 350 W, t = 2 hrs	Chung C., Chung T. & Yang T., (2008). Rapid Synthesis of Titania Nanowires by Microwave-Assisted Hydrothermal Treatments. *Ind. Eng. Chem. Res.*, Vol. 47, No. 7, pp. 2301-2307.
TiO_2 Photocatalysts	T = 393, 423 K, t = 15, 30, 60 min	Addamo M., Bellardita M., Carriazo C., Paola A., Milioto S., Palmisano L. & Rives V., (2008). Inorganic gels as precursors of TiO_2photocatalysts prepared by low temperature microwave or thermal treatment. *Appl. Catalysis B: Environmental*, Vol. 84,pp. 742-748.
Titanate Nanowires	T = 210 ˚C, P = 350 W, t = 5 hrs	Chang M., Chung C., Deka J., Lin C. & Chung T., (2008). Mechanical properties of microwave hydrothermally synthesized titanate nanowires. *Nanotechnology*, Vol. 19, pp. 025710.
Mesoporous$AlPO_4$-5 (AFI) Zeotype	T = 180 - 200 ˚C, t = 0.5 - 2 hrs	Utchariyajit K. &Wongkasemjit S., (2008). Structural aspects of mesoporous $AlPO_4$-5 (AFI) zeotype using microwave radiation and alumatrane precursor. *Microporous and Mesoporous Materials*, Vol. 114, pp. 175-184.
Zn,Al-CO_3 Layered Double Hydroxides	T = 100, 125 ˚C, t = 30, 60, 180, 300 min	Benito P., Guinea I., Labajos F., Rocha J. & Rives V., (2008). Microwave-hydrothermally aged Zn,Alhydrotalcite-like compounds: Influence of the composition and the irradiation conditions. *Microporous and Mesoporous Materials*, Vol. 110, pp. 292-302.
Ni-Al, Zn-Al Hydrotalcite	T = 100, 125, 150, 175 ˚C, t = 5 - 300 min	Benito P., Herrero M., Barriga C., Labajos F. & Rives V., (2008). Microwave-Assisted Homogeneous Precipitation of Hydrotalcites by Urea Hydrolysis. *Inorg. Chem.*, Vol. 47, No. 12, pp. 5453-5463.

Synthesis, Passivation and Stabilization of Nano (particles, rods & wires)	P = 650 W (60%), t = 10 sec to mins	Abdelsayed V., Panda A. B., Glaspell G. P. & El-Shall M. S., (2008). Synthesis, Passivation and Stabilization of Nanoparticles, Nanorods and Nanowires by Microwave Irradiation. *ACS Symposium Series*, Vol. 996, pp. 225-247.
$LaPO_4$:RE (RE=Ce,Tb,Eu) and In_2O_3:Sn (ITO)	T = 300 ˚C, t = 0 - 30 sec	Buhler G., Stay M. &Feldmann C., (2007). Ionic liquid based approach to nanoscale functional materials. *Green Chemistry*, Vol. 9, pp. 924-926.
$ZnIn_2S_4$ Submicrospheres	T = 200 ˚C, t = 10 min	Hu X., Yu J., Gong J. &Quan Li., (2007). Rapid Mass Production of Hierarchically Porous $ZnIn_2S_4$Submicrospheres via a Microwave-SolvothermalProcess.;*Cryst. GrowthDes.*,Vol. 7, No. 12, pp. 2444-2448.
Co^{2+} in Layered Double Hydroxides	T = 100, 125 ˚C, t = 10, 30, 60, 180 min	Herrero M., Benito P., Labajos F. &Rives V., (2007).Stabilization of Co^{2+} in layered double hydroxides (LDHs) by microwave-assisted ageing. *J. Solid State Chem.*, Vol. 180, pp. 873-884.
MFI Zeolite Membranes	T = 80, 120 ˚C, P = 250, 400 W, t = 60, 90 min	Motuzas J., Heng S., Lau P., Yeung K.,Beresnevicius Z. &Julbe A., (2007). Ultra-rapid production of MFI membranes by coupling microwave-assisted synthesis with either ozone or calcination treatment. *Microporous and Mesoporous Materials*, Vol. 99, pp. 197-205.
Titanate Nanotubes	T = 110, 130, 150, 175 ˚C, P = 70, 200, 400, 700 W, t = 1.5, 3, 6, 12 hrs	Ou H., Lo S. &Liou Y., (2007).Microwave-induced titanate nanotubes and the corresponding behavior after thermal treatment. *Nanotechnology*, Vol. 18, pp. 175702.
α-Fe_2O_3 Nanoarchitectures	T = 120 - 220 ˚C, t = 10 min	Hu X., Yu J. &Gong J., (2007). Fast Production of Self-Assembled Hierarchical α-Fe_2O_3Nanoarchitectures. *J. Phys. Chem. C*, Vol.111 , No. 30, pp. 11180-11185.
Nanosized Cobalt Oxides	T = 100 ˚C, t = 10, 30, 60, 180, 300 min	Herrero M., Benito P., Labajos F. & Rives V., (2007). Nanosize cobalt oxide-containing catalysts obtained through microwave-assisted methods. *Catalysis Today*, Vol. 128, pp. 129-137.
Nanorods and Nanoplates of Rare Earth Oxides	P = 650 W (70%)	Panda A. B., Glaspell G. & El-Shall M. S., (2007). Microwave Synthesis and Optical Properties of Uniform Nanorods and Nanoplates of Rare Earth Oxides. *The Journal of Physical Chemistry B Letters*. Vol. 111, pp. 1861-1864.

$AlPO_4$-18 Films	T = 150 - 200 ˚C, P = 1 kW	Vilaseca M., Yagüe C., Coronas J. &Santamaria J., (2006).Development of QCM sensors modified by $AlPO_4$-18 films. *Sensors and Actuators B: Chemical*, Vol. 117, pp. 143-150.
Modification of TiO_2	T = 145 ˚C, P = 1.2 kW, t = 5 -360 min	Wilson G., Matijasevich A., Mitchell D., Schulz J. &Will G., (2006). Modification of TiO_2 for Enhanced Surface Properties: Finite Ostwald Ripening by a Microwave Hydrothermal Process. *Langmuir*, Vol. 22, No. 5, pp. 2016-2027.
Nano-sized Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$)	T = 85 - 300 ˚C, P = 250 - 650 W, t = 4 min	Han J., Song H., Saito F. &Lee B., (2006). Synthesis of high purity nano-sized hydroxyapatite powder by microwave hydrothermal method. *Mater. Chem. Phys.*, Vol. 99, No. 2-3, pp. 235-239.
Ba-exchanged zeolite A	T = 150, 200, 230 ˚C, P = 1kW, t = 40, 60, 120 min	Ferone C., Esposito S. &Pansini M., (2006). Microwave assisted hydrothermal conversion of Ba-exchanged zeolite A into metastable paracelsian. *Microporous andMesoporous Materials*, Vol. 96, pp. 9-13.
Nanocrystalline Anatase TiO_2	P = 950 W, t = 2 - 6 min	Murugan A., Samuel V. &Ravi V., (2006).Synthesis of nanocrystallineanatase TiO_2 by microwave hydrothermal method. *Materials Lett.*,Vol. 60, pp. 479-480.
Semiconductor Rods and Wires	P = 650 W (60%), t = 30 sec to 3 mins	Panda A. B., Glaspell G. & El-Shall M. S., (2006). Microwave Synthesis of Highly Aligned Ultra Narrow Semiconductor Rods and Wires. *J. Am. Chem. Soc.*, Vol. 128, No. 9, pp. 2790-2791.
TiO_2 Nanocrystals	P = 650 W (33%), t = 30 mins	Glaspell G., Panda A. B. & El-Shall M. S., (2006). Reversible paramagnetism to ferromagnetism in transition metal-doped TiO_2 nanocrystals prepared by microwave irradiation. *Journal of Applied Physics*, Vol. 100, pp. 124307-12412.
Silicalite-1 Seeds	T = 70 - 180 ˚C, P = 250, 400 W, t = 10 - 150 min	Motuzas J., Julbe A., Noble R. D., Guizard C., Beresnevicius Z. J. &Cot D., (2005).Rapid synthesis of silicalite-1 seeds by microwave assisted hydrothermal treatment. *Microporous and MesoporousMaterials*,Vol. 80, pp. 73-83.
Coaxial Ag/C Nanocables	T = 200 ˚C, t = 20 min	Yu J., Hu X., Lib Q. &Zhang L., (2005).Microwave-assisted synthesis and in-situ self-assembly of coaxial Ag/C nanocables. *Chem. Commun.*, pp. 2704-2706.

M-Doped ZnO (M = co, Cr, Fe, Mn, Ni)	P = 650 W (33%), t = 10 min	Glaspell G., Dutta P. & Manivannan A., (2005). A Room-Temperature Synthesis of M-Doped ZnO (M = Co, Cr, Fe, Mn & Ni), *Journal of Cluster Science*, Vol. 16, No. 4, pp. 523-536.
Au and Pd Nanoparticle Catalysts	P = 650 W (33%), t = 10 mins	Glaspell G., Fuoco L. & El-Shall M. S., (2005). Microwave Synthesis of Supported Au and Pd Nanoparticle Catalysts for CO Oxidation. *The Journal of Physical Chemistry B Letters*, Vol. 109, pp. 17350-17355.
$AlPO_4$-5	T = 180 ˚C, P = 500 W, t = 17 min	Lin C., Dipre J. &Yates M., (2004).Novel Aluminum Phosphate-5 Crystal Morphologies Synthesized by Microwave Heating of a Water-in-Oil Microemulsion. *Langmuir*, Vol. 20,No. 4, pp. 1039-1042.
Crystallization of Zeolite A	P = 180, 360, 900, 1260 W, t = 10, 30, 40, 50 min P = 360, 900 W, t = 5,10, 20,30, 50 min P = 360 W, t = 15, 30, 40 min	Bonaccorsi L. &Proverbio E., (2003).Microwave assisted crystallization of zeolite A from dense gels. *J. Cryst. Growth*, Vol. 247, No. 3-4, pp. 555-562.
Sodalite/αAl_2O_3 Membranes	T = 160 - 200˚C, P = 250 W, t = 15 - 180 min	Julbe A., Motuzas J., Cazevielle F., Volle G. &Guizard C., (2003). Synthesis of sodalite/αAl_2O_3 composite membranes by microwave heating. *Separation andPurification Technology*, Vol. 32, pp. 139-149.
$BaTiO_3$ Film	T = 100 ˚C, t = 5, 10, 15, 30, 60 min	Lee J., Kumagai N., Watanabe T. &Yoshimura M., (2002). Direct fabrication of oxide films by a microwave-hydrothermal method at low temperature. *Solid State Ionics*, Vol. 151, pp. 41-45.
Titanium Silicates	T = 175 ˚C, P = 800 W	Prasad M., Kamalakar G., Kulkarni S., Raghavan K., Rao K., Prasad P. &Madhavendra S., (2002). An improved process for the synthesis of titanium-rich titanium silicates (TS-1) under microwave irradiation. *Catalysis Commun.*,Vol. 3, pp. 399-404.

Hydrodechlorination of Chlorobenzene on Pd/Nb_2O_5 Catalysts	T = 200 ˚C, P = 650 W, t = 5 min	Gopinath R., Rao K., Prasad P., Madhavendra S., Narayanan S. &Vivekanandan G., (2002).Hydrodechlorination of chlorobenzene on Nb_2O_5-supported Pd catalysts: Influence of microwave irradiation during preparation on the stability of the catalysts. *J. of Mol. Catalysis A: Chemical*, Vol. 181, pp. 215-220.
Co-, Co-Zn and Ni-Zn Ferrite Powders	T = 120, 150, 180, 200 ˚C, t = 30 min	Kim C. K., Lee J. H., Katoh S., Murakami R. &Yoshimura M., (2001). Synthesis of Co-, Co-Zn and Ni-Zn ferrite powders by the microwave hydrothermal method. *Mater. Res. Bull.*, pp. 2241-2250.
Uniform Pure and Dye-Loaded $AlPO_4$-5 Crystals	T = 145 - 170 ˚C, P = 1 kW, t = 5 - 60 min	Ganschow M., Schulz-Ekloff G., Wark M., Wendschuh-Josties M. &Wohrle D., (2001). Microwave-assisted preparation of uniform pure and dye-loaded $AlPO_4$-5 crystals with different morphologies for use as microlaser systems. *J.Mater. Chem.*, Vol. 11, pp. 1823-1827.
Synthesis of $AlPO_4$-5	T = 180 - 190 ˚C, P = 900 W, t = 8 min	Braun I., Schulz-Ekloff G. &Lautenschläger W., (1998).Synthesis of AlPO4-5 in a microwave-heated, continuous-flow, high-pressure tube reactor. *Microporous andMesoporous Materials*, Vol. 23, pp. 79-81.

Table 1. Summary of selected materials prepared using microwave heating with conditions and apporiate references listed.

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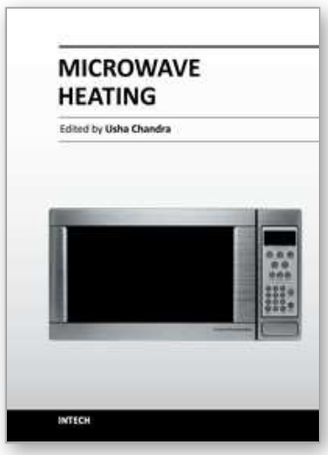

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