Study the Heating Mechanisms of Temperature Controlled Microwave Closed System (TCMCS)

F. Kormin, N. H. Abdurahman, R. M. Yunus, Rivai

Abstract— The efficiency of temperature controlled microwave closed system (TCMCS) on heating mechanism was investigated. Heating mechanism of TCMCS was studied on plant material (Nephrolepis biserrata) immersed in water, acetone, acetonitrile and ethanol at different microwave power level. The rising temperature of TCMCS was influenced by their dielectric, physical and heating properties of the solvents. After reaching the boiling point, the solvent density decreased and this factor affected in reducing of dielectric properties. Water which has lower mass heat capacity took time compared to acetone, acetonitrile and ethanol. The rate of heat generation and penetration depth was calculated. The decreasing of heat generation within radiation time is due to the decreasing dielectric properties of solvents with regards to temperature increase. The penetration depth at high microwave power is higher than at lower microwave power. As a polar solvent, water was efficiently absorb microwave energy and leads to efficient heating at 700Watt followed by 560>420>280>140Watts. This provides a qualitative understanding of microwave heating mechanism.

Index Terms— Dielectric Properties, Heating Mechanism, Penetration Depth, TCMCS, Volume Rate Of Heat Generation.

I. INTRODUCTION

Microwave-enhanced chemistry is based on the efficient heating of materials by “microwave dielectric heating” effects. This phenomenon is dependent upon the ability of a specific material or solvent to absorb microwave energy and convert it into heat. The ability of a material to interact with microwaves is dependent on the dielectric constant ($\varepsilon'$) and dielectric loss ($\varepsilon''$). The dielectric constant is a measure of a sample’s ability to obstruct the microwave energy as it passes through, and the loss factor measures the sample’s ability to dissipate that energy. The word “loss” is used to indicate the amount of input microwave energy that is lost to the sample by being dissipated as heat (Mandal et al., 2007). Knowledge of the dielectric properties of the material being heated is of great importance in microwave processing.

The effect of microwave energy is strongly dependent on the nature of the solvent. The solvent should generally be capable of absorbing the microwave energy. Apart from absorbing the energy, the solvent must be able to convert this energy into heat, so the efficiency of the conversion process is dependent on the dielectric factor loss. Dielectric properties of the solvent towards microwave heating play an important role in microwave heating. Solvents with different dielectric properties, when exposed to microwave radiation, will have a different response to it. Ordinarily, the higher value of dielectric constant, the more efficient the solvent absorbs the microwave energy and heated more efficiently. Solvents with very low dielectric constant and loss factors cannot couple with microwave oscillation efficiently. Therefore, will not absorb microwave energy (transparent to microwave energy). Since dielectric loss and dielectric constant are temperature dependent, thus the dissipation factor (loss parameters of microwave applications) will increase with temperature. Therefore, the heating rate for these polar or nonpolar solvents will increase during microwave heating (Perreux and Loupy, 2001) probably by limiting the formation of “boiling nuclei” (Baghurst and Mingos, 1992). Mingos sand Barghust (1991) has claimed that the different rate acceleration can be contributed to the solvent superheating induced by microwave irradiation. The commercial microwave involves the safety issue of increased temperature and pressure leading to burning of the sample and rupture of the container. The potential for commercial microwave heating to be applied in a process is dependent upon the dielectric properties to the target material. Such properties are not fully understood. Not all materials possess the ability to absorb microwave radiation, which, although a limiting factor in some applications, can be advantageous in others (Appleton et al., 2005). Therefore, for a reason of safely and in order to ensure optimum reproducibility, study in this unequal heating of samples in the microwave field must always be taken into
consideration. More research is needed to improve the understanding of microwave extraction mechanism, remove technical barriers, improve the design and scale up of the novel extraction systems for their better industrial applications. Accordingly, a primary goal of our novel microwave extraction research is to develop of methods that are more efficient and highly representative extracts.

Temperature is an important factor in conventional extraction methods, especially in the case of botanical extractions. But, study on the extraction temperature is quite rare in microwave extraction technique as it is not easy to control the temperature in the microwave oven. The principle of heating using microwaves is based on the direct effect of the waves on molecules by ionic conduction and dipole rotation. The plant matrix was immersed in high dielectric constant solvent which is rapidly heated by microwaves, disrupting the cellular structure and releasing the desired components into the surrounding medium. Thus, the temperature controlled microwave closed system (TCMCS) with fluid stirring device is a relatively provide new extraction technique that delivers microwave energy rapidly to a total volume of the solvent and solid plant matrix in controlled temperature. Therefore, efficiency of TCMCS parameters comprises various solvent and microwave power on volume rate of heat generation and penetration depth of TCMA of plant extract was studied.

II. MATERIALS AND METHODS

A. Microwave Oven Modification

Figure 1 shows the block diagram of temperature controlled microwave closed system (TCMCS). The TCMCS was design with temperature controller and fluid seal stirring system. The temperature of extraction of the microwave system was controlled by electrically capillary thermometer. The responsive probe of electrically capillary thermometer was inserted into a target extracted materials in a microwave oven. The electrically capillary temperature probe was controlled by contactor which adapted to provide a signal to interrupt the application of microwave power upon reaching a predetermined temperature. Therefore the operation temperature can be controlled in low temperature on the extraction of thermo sensitive compounds and also can be controlled in unlimited temperature where applicable to heat the extraction solvent above its atmospheric boiling point. The timer was counted upon the temperature reached the predetermined temperature to control the time of extraction. Table I. shows a pulse and rest time of TCMCS at complete cycle on each microwave power level.

![Diagram](image-url)

**Fig.1. Block diagram of temperature control microwave closed system (TCMCS)**
In this study, *Nephrolepis bisperrata* frond (NBF) was chosen as a plant material. The dried NBF were ground by Panasonic MX896TM grinder. The grounded materials were passed through a stainless steel sieve with a pore size of 0.3 mm (Impact Laboratory Test Sieve 850 MIC aperture). The sieved plant materials (16.5 g) were weighed and transferred into a reaction vessel where 500 mL of solvent was added.

**B. Conversion of Microwave Energy into Heat**

The thermal properties of materials are also an important factor in the study of microwave heating. As a material is heated by microwaves, the mass heat capacity ($C_p$) of the microwave-absorbing material is required to determine the amount of energy absorbed by the material. The mass capacity is the quantity of heat required to raise the temperature of a given mass by 1°C. The energy absorbed produces a rise in temperature, $\Delta T$. If a quantity of energy delivered for a unit of time ($P$) absorbed by a substance (power density) in the microwave cavity may be expressed in the following relation (Kingston and Jessie, 1988). The following equation (1) is used to calculate the energy requirements:

$$
P_{absorbed} = \frac{\Delta T \times K \sum C_p m_i}{t}
$$

(1)

Where:

- $P$ = the apparent power absorbed by the sample in watt (W) (joule/sec)
- $K$ = the conversion factor for thermochemical sec-1 to watt (K=4.184)
- $C_p$ = the heat capacity, thermal capacity or specific heat [cal/(g*°C)] of water (heat capacity of water at 25°C is 0.9997)
- $m$ = the mass of the water sample in gram (g)
- $T$ = the final temperature minus the initial temperature (°C)
- $t$ = the time in seconds (s)

The one-dimensional unsteady state heat conduction equation with heat generation may be used to describe microwave heating of material (Taher and Farid, 2001) undergoing to the materials as shown in equation (2):

$$
\rho_{mix} C_{p,mix} \frac{\partial T}{\partial t} = \frac{\partial}{\partial y} \left( \sigma_{mix} \frac{\partial T}{\partial y} \right) + P_{absorbed}
$$

(2)

where

<table>
<thead>
<tr>
<th>Pulse time</th>
<th>Rest time</th>
<th>Cycle time</th>
</tr>
</thead>
<tbody>
<tr>
<td>140 Watt</td>
<td>3.38</td>
<td>26.63</td>
</tr>
<tr>
<td>280 Watt</td>
<td>4.64</td>
<td>25.37</td>
</tr>
<tr>
<td>420 Watt</td>
<td>11.49</td>
<td>18.55</td>
</tr>
<tr>
<td>560 Watt</td>
<td>18.49</td>
<td>11.58</td>
</tr>
<tr>
<td>700 Watts</td>
<td>30.00</td>
<td>0</td>
</tr>
</tbody>
</table>
\[ \rho_{\text{mix}} = \text{bulk density of dope solution (gm.cm}^{-3}\text{)} \]

\[ C_{p,\text{mix}} = \text{specific heat capacity of mixture of material (cal.g}^{-1}.\text{.C}^{-1}\text{)} \]

\[ \frac{\partial T}{\partial t} = \text{raise of temperature due to microwave radiation (}^{\circ}\text{C.sec}^{-1}\text{)} \]

\[ \sigma_{\text{mix}} = \text{thermal conductivity of mixture of material (cal.cm}^{-1}.\text{.C}^{-1}\text{)} \]

\[ y = \text{location of sample} \]

Equation (3) comprises of three terms, convective heat transfer, radiation heat due to microwave and conductive heat in the sample respectively. The effect of radiation is very small as well as the convective term, since the sample container (glass) has a low dielectric constant. Therefore, it’s heat generation \( q_{\text{MW}} \) (cal/sec cm\(^3\)) is assumed to be negligible. The volume rate of heat generation with respect to rate of temperature rise (\(^{\circ}\text{C}\).sec\(^{-1}\)) would be given by equation (3):

\[ q_{\text{mv}} = \rho_{\text{mix}} C_{p,\text{mix}} \frac{dT}{dt} \quad (3) \]

\( q_{\text{MW}} \) is a constant depending on the electric field and mobility of the materials at fix location.

The \( \varepsilon' \) is the measure of the material’s ability to absorb microwave energy and the \( \varepsilon'' \) represents the efficiency of converting microwave energy into heat. The basic dielectric relations are as follows (Hoekstra and Delaney, 1974).

\[ \varepsilon = \varepsilon' - j\varepsilon'' \quad (4) \]

Where:

- \( \varepsilon \) = the complex dielectric constant
- \( \varepsilon' \) = the relative dielectric constant
- \( \varepsilon'' \) = the relative dielectric loss
- \( J \) = imaginary number (\(\sqrt{-1}\))

\[ \varepsilon'' = \frac{\sigma}{\omega\varepsilon'} \quad (5) \]

Where:

- \( \varepsilon' \) = the dielectric constant of free space (8.8 x 10\(^{-12}\) Farad/m)
- \( \omega \) = angular frequency (Hz)
- \( \sigma \) = electrical conductivity (S/m)

\[ \varepsilon'' = \varepsilon' \tan \delta \quad (6) \]

Where \( \tan \delta \) is the dissipation factor or loss tangent, which represents the ability of the material to absorb microwave energy and pass it on in the form of heat to other molecules. Thus, both \( \varepsilon'' \) and \( \tan \delta \) determines the amount of heat that will be generated when a solvent is subjected to microwave.

When microwave energy penetrates a sample, the energy is absorbed by the sample at a rate dependent upon its dissipation factor. Penetration is considered infinite in materials that are transparent to microwave energy and are considered zero in reflective materials such as metals. The dissipation factor is a finite amount for absorptive
samples. Because the energy is quickly absorbed and dissipated as microwaves pass through the sample, the greater
the dissipation factor of a sample, the less the penetration of the microwave energy at a given frequency. A useful
way to characterize penetration is by the half power depth for a given sample at a given frequency. The half-power
depth is defined as that distance from the surface of a sample at which the power density is reduced to one-half that
at the surface. The half-power depth varies with the dielectric properties of the sample and approximately with the
inverse of the square root of the frequency. The dielectric constant (ε) and dielectric loss (δ) of water are given by
the following equations (7) and (8):

\[
E_{r,w} = 85.215 - 0.33583T \quad (7)
\]

\[
E_{r,w} = 320.658T^{0.268} \quad (8)
\]

Which are the least-fit equations to the data given by von Hippel (1954). The temperature, T is in °C. Looyenga’s
formula for polar solvent’s mixture is as follows:

\[
Y_{s,m} = \left[ Z_{s1} + V_{s2} \left( Z_{s1}^{0.3} + Z_{s2}^{0.3} \right) \right]^{3} \quad (9)
\]

Where Y can be a solvent mixture, where subscription m represents a mixture of materials and s represents
solvents. The dielectric loss (δ) or tangent loss (tan δ) of mixture of materials was given by Raju (2003):

\[
\tan \delta_{s,m} = \frac{\phi_{s1}\varepsilon_{s,1}^{1.2}\tan \delta_{s,1} + \phi_{s2}\varepsilon_{s,2}^{1.2}\tan \delta_{s,2}}{\phi_{s1}\varepsilon_{s,1}^{1.2} + \phi_{s2}\varepsilon_{s,2}^{1.2}} \quad (10)
\]

Where, φ is mole fraction. The dissipation factor or dielectric loss tan for the materials in a mixture of two solvents
may be approximated by the equation:

\[
\tan \delta_{s,m} = \frac{\phi_{s1}\varepsilon_{m1}^{1.2}\tan \delta_{s,1} + \phi_{s2}\varepsilon_{m2}^{1.2}\tan \delta_{s,2}}{\phi_{s1}\varepsilon_{m1}^{1.2} + \phi_{s2}\varepsilon_{m2}^{1.2}} \quad (11)
\]

Usually, most materials are measured in weight grams by so it is often necessary to convert the weight fraction (w)
to volume fraction (V) of the components upon dealing with multicomponent materials (Bicerano, 2002). Thus, the
following equation is required for conversion:

\[
K_f \phi_m = V_n = \left( \frac{w_i}{\rho_i} \right) \times \sum_{j=1}^{n} \frac{\rho_j}{w_j} \quad (12)
\]

Where, ρ is the density of each material; w is a weight fraction of each material. Subscription n may be a
material, a solvent or a mixture of solvents.

A large penetration depth indicates that radiation poorly is absorbed while short penetration depth means that
surface heating predominates. Depth of penetration of microwaves is dependent on dielectric properties of material
composition and the frequency, with decreasing frequency being more penetrated (Metaxas and Meredith, 1983).
The penetration depth is used to denote the depth at which the power density has decreased to 37 % of its initial
value at the surface. Material with higher loss factor εr” show faster microwave energy absorption. The power
density will decrease exponentially from the surface to the core region.

As a wave progresses into a dielectric-heating sample, its amplitude diminished owing to absorption of power as
heat in the material. In the absence of reflected waves through the material, the field intensity and its associated
power flux density fall exponentially with distance from the surface. Because the power absorbed in an element
volume of material is proportional to power flux density flowing through it, the power dissipation also falls
exponentially from the surface. The rate of decay of the power dissipation is a function of both the relative

\[
E_{r,w} = 85.215 - 0.33583T \quad (7)
\]

\[
E_{r,w} = 320.658T^{0.268} \quad (8)
\]
permittivity, dielectric constant ($\varepsilon$) and loss factor ($\varepsilon''$). The penetration depth, $D_p$ is defined as the depth into material at which the power flux has fallen to $1/e$ (equal to 0.368) of its surface value and its readily shown to be given by equation (13):

$$D_p = \frac{\lambda_0}{2\pi \sqrt{2\varepsilon}} \frac{1}{\sqrt{\left(1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2\right)^{0.5}}} - 1$$

(13)

Where $\varepsilon'' < \varepsilon'$, equation (13) can be simplified to equation (14), with an error up to 10%.

$$D_p = \frac{\lambda_0}{2\pi \varepsilon}$$

(14)

Where $\lambda_0$ is the wavelength of the microwave energy in free space (32.76 cm at 915 MHz and 12.24 cm at 2450 MHz) (Von Hippel, 1954). The penetration depth is a very important parameter for a sample because it gives an immediate first-order indication of the heat distribution within it. Note that $D_p$ does not mean that heating at a depth exceeding the penetration depth. The heat dissipates in the layer bounded by the surface and the plane at depth $D_p$ is only 63.2% of the total, the balance being dissipated in the material at depth greater than $D_p$ (Meredith, 1998).

### III. RESULTS AND DISCUSSION

#### A. Microwave Power Absorbance

Based on the measurements of rising temperature of every 20 sec for polar solvents: water, acetone, acetonitrile, and ethanol, the microwave power absorbance was calculated and tabulated in Figure 2. Microwave power absorbed of each solvent in the microwave oven was determined experimentally by measuring rate of the temperature rise after heating known weight of each solvent at every interval time of each cycle pulse using equation (1). The increasing order of % absorbance value is as follows: water > acetonitrile > ethanol > acetone. It was observed that the power absorbance of water is higher as compared to acetonitrile, acetone and ethanol due to its high dielectric constant and dipolar moment.

Microwave heating is also referred to as dielectric heating (Oespchuck, 1984). The ability of water to absorb energy in a microwave cavity is related to the high dielectric properties of the water. The molecules or atoms comprising the dielectric exhibit a dipole movement. This movement generates friction inside the dielectric and the energy is dissipated subsequently as heat (Kelly and Rowson, 1995). The interaction of dielectric properties of solvent with electromagnetic radiation in the microwave range results in energy absorbance. This depends on the relaxation times of the molecules in the material, which in turn depends on the nature of the functional groups, the volume of the molecule (Gabriel et al., 1998) and number of carbon atoms in the molecule. Liao (2002) and Yilmaz (2002) were found that, a number of carbons in the molecule increases, the dielectric constant decreases and the result was accordance with our finding.

Figure 2 also shows the experimental results of % absorbance for five microwave power level. The microwave powers absorbance of water increases with increased microwave power levels. A similar phenomenon was observed in acetone acetonitrile and ethanol. In such cases, equalization of thermal energy due to conduction during microwave power-off periods tends to result in a more uniform temperature profile across the sample. The microwave power absorbance by the materials is very much related to the cycle pulse. At 700 watts, continuous heating occurs and this means that materials are continuously exposed at nonstop of radiation time. The higher microwave power level resulted the longer pulse time to microwaves can see at Table I. Therefore, it would affect the movement state and reaction activity of the various molecules in the reaction system of solution. Consequently, at 140, 280 and 420 Watts due to short pulse time, the temperature increments are low which resulted low % absorbance. Similar findings were also reported by Zhu et al. (2003), they has found that the rise of temperature and microwave power absorbed to the materials depend upon radiation time and it is establish that at a high power level.
B. Heating Mechanism of TCMCS under Different Solvents

The heating mechanisms of microwave and conventional heating are different. In microwave heating, time-temperature profiles within the product are mainly caused by internal heat generation due to absorption of electrical energy from the microwave field, and then heat is transferred by conduction, convection, and evaporation (Mudgett, 1982). Therefore, different solvent with dissimilar dielectric, physical and thermal properties at different microwave frequencies and exposure time revealed a dissimilar response on their polarity, dipole strength, and composition within heating.

In order to investigate the effect of dielectric properties in TCMCS heating mechanism, the relation between rising temperatures with microwave radiation time (at 20 sec, 40 sec, 60 sec, 80 sec, 100 sec, 120 sec, 180 sec, 240 sec and 300 sec.) was studied. Figure 3 was plotted to present the relation between the microwave heating rate and radiation time of plant material immersed in water, acetone, acetonitrile, and ethanol at 700 Watts. As illustrated, the temperature of various solvent increased linearly but after 100 sec acetone, acetonitrile and ethanol was inversely proportional with time of heating. The rising temperature was influence by their dielectric properties of the solvents, which is reflecting on solvent polarity and the ability of microwave to interact with the materials and transfer the microwave energy (Chan and Chen, 2002; Bjorndalen and Islam, 2004). The interaction of dielectric properties of solvent with electromagnetic radiation in the microwave range consequences with energy absorbance. If the solvent molecule is not able to absorb microwave energy, there will be no heating and the temperature not rise up.

In this study, each solvent having a good dielectric properties value means the solvent molecules become energized during heating, thus resulting in higher reaction rates (Strauss and Trainor, 1995; Fre’re et al.2001). However, the dielectric properties also influence by temperature, moisture content and density (Metaxas and Meredith, 1993). It has been shown in the result, after the solvent reaching their boiling points, the temperature was reduced. According to Uematsu and Franck (1980), the lessening of dielectric constant value by rising of temperature can be explained by decreasing of solvent density during heating extension.

It has been examined in Figure 3, the physical properties such as boiling points and density of the solvent were affected with increasing and reducing temperature within time. The lower density of solvent like acetone, ethanol and acetonitrile was increased rapidly within first 60 seconds as compared to water which is having higher density. In addition, water which is highly boiling points was taking time to reach that point as compared to other solvent
which is reaching constant temperature less than 120 seconds. According to Ryynänen (2002), the physical properties such as water volume fraction, viscosity, mass heat capacity and thermal conductivity, was greatly influenced the rising temperature of the emulsion but slightly influence on dielectric properties of emulsion. In this study, it can be seen that the temperature elevation is also much dependent on the thermal properties of the solvent. As shown in the Figure 3, the mass heat capacity (cp) of the solvent influence the amount of energy absorbed during microwave heating. The mass heat capacity is the amount of energy required to raise one degree of temperature of a given mass of material. It was found that the acetone which is having highest value of mass heat capacity easier to response to microwave heating at opening time followed by acetonitrile, ethanol and water. As a result, in microwave heating, the lower mass heat capacity solvent will taking time to rise a temperature as compared to higher mass heat capacity.

The magnitude of the solvent dipole moment is another main factor that correlates with the microwave heating characteristic (Neas and Collins, 1988) of protic and aprotic polar solvent. Inspection of dipole moments values suggest that acetone and acetonitrile with lowest dipole moments will rotate easily when exposed to an alternating electric field of microwave energy (Bottcher, 1952) as compared to water and ethanol. This oscillation produced collisions with surrounding molecules, then energy was transferred with subsequent heating (Saoud, 2004).

![Figure 3. Rising temperature of various solvent versus radiation time](image)

C. Volume Rate of Heat Generation

In this study, the volume rate of heat generation was calculated to support the previous result. In this study, the TCMCS performance assisted were investigated at 700 Watts. The heat generation is directly related to the rate of temperature increase, density and heat capacity of the solvents. Since the rate of heat generated by the materials inside the microwave cavity is instantly interrelated to the aptitude of the molecules to align itself with the frequency of the applied field. Therefore, the rate of heat generation of solvents can be calculated using equations (3).
As observed in Figure 4, the result of volume of heat generation of all of the solvents increased almost linearly with the increased of exposure time then as the exposure time was increased further, the volume rate of heat generation decreased and in certain cases approached plateau condition. The average value from 10 point of reading shows water is the highest, which is 1.59 Watts/mL and the lowest is 0.84 Watts/mL of acetone. It is observed that each solvent, exhibits temperature increments according to the dipole and high dielectric constant asset value. The decreasing of heat generation within radiation time is due to the decreasing dielectric properties of solvents with regards to temperature increase. The polarity of solvent decreases with increasing temperature, which resulted decreases of the ability of water to absorb microwave energy and affected the dipole rotation and alignment of water molecules in the microwave field. The dielectric property which is attributed to the solvent polarity is dependent on mobility of the dipoles within the structure (Rahmat, 2002). The dielectric properties which are affected the ability of samples to absorb and dissipate the microwave energy are influenced by heating temperature (Chan and Chen, 2002; Saound, 2004). In general, the ability of material to dissipate energy changes as the property of the material changes.

As a strongly high dipolar moment, water can efficiently absorb the microwave energy and transform it into thermal energy, leading to the efficient heating of the sample. Apart from absorbing the energy, the solvent also must be able to convert this energy into heat. The capability of a water to convert microwave energy into heat at a given frequency and temperature is determined by the so-called loss tangent (tan δ), expressed as the quotient, dielectric loss/dielectric constant (\( \varepsilon'' / \varepsilon' \)) were describing the ability of molecules to be polarized by the electric field (Herrero et al., 2008). As a conclusion, solvent with a high tanδ at the standard operating frequency of a microwave synthesis reactor (2.45 GHz) is required for good absorption and, consequently, for efficient heating. Acetone has a lowest dielectric constant of among all selected solvents, thus attributing to low microwave energy absorbed.

![Fig.4. Volume rate of heat generation vs radiation time](image)
D. Penetration Depth

In this study, the penetration depth was calculated to support the previous result. The TCMCS performance were also investigated at 700 Watts. Penetration depth is an effective and convenient measure to compare the relative microwave absorbing characteristics of samples and to explain the effect of the dielectric properties and geometry on microwave heating. A large penetration also indicates that radiation is poorly absorbed while a short penetration depth means that surface heating predominates (Saoud, 2004). The penetration depth also affected by microwave frequency (Hoekstra and Delaney, 1974). High frequency has a short wavelength which corresponds to a smaller penetration depth (Ratanadecho, 2006). In this study, the penetration depth was calculated for TCMAE of plant material in water, acetone, acetonitrile and ethanol as a solvent. Figure 5 shows the result of penetration depth are as follows water>ethanol>acetonitrile>acetone. The result was accordance to Palacios et al., (1996) which found that ethanol has the shorter penetration depth than water, which demonstrated that the microwave heating rate of ethanol is higher than water.

![Fig.5. Penetration depth of various solvent vs radiation time](image)

E. Heating Mechanism of TCMCS under Different Microwave Power Level

Figure 6 presents the rising temperature of TCMCS in different microwave power level (140, 280, 420, 560 and 700 Watts). Temperatures of all conditions were measured across the vertical mid-plane of the reaction vessel. In this study, the temperature of water was recorded from microwave oven at every 60sec in 16 intervals of time. The temperature measurements were made using the type-K thermocouple probe connected to a digital meter. The rising temperature results show the TCMCS temperature at 700 watts increase rapidly as compared to another power level. In general, the increasing order of rising temperature is as follows: 700>560>420>280>140 watts. At
700, 560, 420 and 280 Watts, the water temperature reached the boiling points (100°C) in 240, 320, 540 and 780 sec respectively. After that the temperature rises in those power levels began to constant and uniform.

![Graph showing temperature rise over radiation time](image)

**Fig. 6. Rising temperature of TCMCS in various power level to radiation time**

It is also observed that the acceleration of microwave absorbed energy inside the water during the initial radiation time is higher at high to low power level where the molecules of the water become aligned and rapidly absorbed microwave's energy due to short of period time. Bicerano (2002) also reported same manners of amorphous polymer in the liquid state because the microwave energies and thus amplitude of the random thermal motions increase at the high pulse level. Moreover, the increment of temperature in water at high power was much stronger than that at the low power level, thus the increment of temperature reaching at high level smoothly and in a very short time.

The accelerated rising temperature by increasing microwave power can be correlated to the direct effects of microwave energy on ionic conduction and dipole rotation which result in power dissipated in a volumetric fashion inside the water which resulted molecular movement and heating. More electromagnetic energy was transferred to the water when the microwave power increased from 140 to 700 watts and improved the raising temperature. At high power level (700 watts), the rate of temperature increases is high and this indicates that the microwave heating is very rapid at continues pulse time. It’s clearly shows that the continuous or longer pulse time has enormous influence on rate of the temperature increase inside the closed system with respect to radiation time (sec) but in short pulse time (140 Watt) water was not reaching their boiling temperature even after 900 sec of reading. In 140 Watts, the pulse is only 3.38 sec as compared to 26.63 rest time in complete cycle of 30.01 sec. That’s mean in 900 sec of radiation time, water only accept 266.27 sec or 29% of microwave pulse. The continuously high rpm stirring tends to result in a more uniform temperature profile across the sample. Since the closed system is transparent thus microwaves can easily penetrate inside the materials at every direction and also the reflection of microwaves inside the cavity helps maintain the uniform temperature profile. The same findings were also reported by Huacai et al. (2006).

**IV. CONCLUSION**

In this study, the new concept of temperature controlled microwave closed system (TCMCS) equipped with a fluid
seal stirrer was successfully built up. The performance of the TCMCS was clearly shown by the demonstration of several solvents. The results observed that in presence of stirring device, the volume rate of heat generation in high dielectric constant solvents show good performances as compared to the low dielectric constant of solvents. Comparison between ethanol and water shows that ethanol has a lower dielectric constant but a higher dielectric loss than water, this indicates that ethanol has lower ability to obstruct the microwave as they pass through, but a higher ability to dissipate the microwave energy into heat. After reach their boiling point the solvent density will be decrease and this factor reflecting in reducing of dielectric properties of solvent. Water with lower mass heat capacity, having higher requirement for microwave energy to be absorbed and consequently, need a time to raise the temperature inside the microwave cavity, therefore, certain criteria must be taken into consideration before solvent or combination of solvents is chosen for microwave extraction in the future study.

REFERENCES