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EDITED BY

Xinwei Bai,
National Energy Technology Laboratory
(Leidos), United States

REVIEWED BY

Jinlong Liu,
Zhejiang University, China
Yifan Deng,
Braskem America Inc., United States

*CORRESPONDENCE

Jamal Chaouki,
jamal.chaouki@polymtl.ca

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Kinetic study of multiphase reactions under microwave irradiation: A mini-review

Kazem Adavi¹, Ahmadreza Amini¹, Mohammad Latifi¹, Jaber Shabaniyan^{1,2} and Jamal Chaouki^{1,3*}

¹Process Engineering Advanced Research Lab (PEARL), Department of Chemical Engineering, Polytechnique Montreal, Montreal, QC, Canada, ²Heirloom, Brisbane, CA, United States, ³Technology Development Cell (Tech-Cell) Department, Mohammed VI Polytechnic University, Ben Guerir, Morocco

Microwave (MW) heating is rapid, selective, and volumetric, and it is a compelling non-conventional heating approach for driving chemical reactions. The effect of MW irradiation on the kinetics of thermal/catalytic reactions is still under debate. A group of researchers reported that the effect of MW heating on reaction kinetics is highlighted through the non-thermal effects of MWs on kinetic parameters and reaction mechanisms in addition to the thermal effect. However, another group attributed the observations to the thermal effect only. In the present work, we summarized and critically synthesized available information in the literature on the subject. It can be concluded that MW heating has solely the thermal effect on gas-solid reactions, and the variations of kinetic parameters are related to the direct and indirect impacts of that. Temperature measurement limitations, physical structure variation, and non-uniform temperature distribution are the primary sources of the discrepancy in previous studies. In ionic liquid-solid reactions, the presence of electromagnetic fields can affect the movement of ions/polar molecules which can be considered a non-thermal effect of MWs. However, the effect of MW absorption by solid/catalyst, and the formation of hot spots must be taken into account to avoid potential discrepancy. Therefore, further theoretical/experimental studies are required to clarify the effect of MWs on liquid-solid reactions. In addition, developing reliable temperature measurement methods and isothermal reaction domain are required for an accurate kinetic study during MW irradiation.

KEYWORDS

microwave irradiation, reaction kinetics, thermal effect, non-thermal effect, temperature measurement, hot spot

Introduction

Compared to conventional heating, microwave (MW) heating is an effective heat supply approach for chemical and material processes. Electromagnetic (EM) properties of materials, including permittivity, permeability, penetration depth, and electrical conductivity play critical roles in absorbing MWs and their conversion to thermal energy (Gupta and Leong, 2007;

TABLE 1 Impact of MW heating on multiphase reactions.

No.	Process	Reactor ID (mm)/ sample amount (g or ml)	Temperature measurement technique	Heating approach	MW effect			Ref.
					Conversion	Selectivity	Reaction temp.	
1	Methane decomposition	22 mm	Pyrometer	Direct	↑ ^a	↑	NA ^b	Bi et al. (1999)
2	Methane oxidation	5 mm	Pyrometer +metallic thermocouple	Direct	↑	↑	↓ ^c	Pham et al. (2020)
3	Methane dry reforming	5–30 mm	Pyrometer	Hybrid	↑	↑	NA	Bai et al. (2022)
4	Methane dehydroaromatization	8 mm	IR	Direct	↑	NA	↓	Durka et al. (2011)
5	Methanol steam reforming	24 mm	Fiber optic probe	Direct	↑	↑	↓	Chen et al. (2022)
7	H ₂ S decomposition	2 g	Metallic thermocouple	Direct	↑	NA	NA	Einaga et al. (2016)
8	CO oxidation	0.1 g	Fiber optic probe	Direct	↑	NA	NA	Ramirez et al. (2020)
9	Propane oxidation	16 mm	Pyrometer	Hybrid	↑	NA	↓	Xia et al. (2020)
10	n-butane oxidation	24 mm	Pyrometer	Hybrid	↑	↑	NA	Hamzehlouia et al. (2018)
11	SO ₂ reduction	20 mm	Metallic thermocouple	Direct	↑	NA	NA	Bai et al. (2019)
12	Ethane valorization	8 mm	Pyrometer	Hybrid	↑	↑	↓	Robinson et al. (2020)
16	Upgrading stranded shale gas to aromatics	10 mm	Pyrometer	Direct	↑	NA	↓	Zhang et al. (2022)
17	Synthesis of phytosterol esters	200 ml	NA	Hybrid	↑	NA	↓	Perry et al. (2002)
18	Sodium tetrahydridoborate (NaBH ₄) hydrolysis	15 mm	NA	Hybrid	↑	NA	↓	Yang et al. (2020)

^a: increasing.

^b: not applicable.

^c: decreasing.

Amini et al., 2021). In the conventional heating of a multi-phase system, an external source supplies heat, and the whole system, including solid particles, gas/liquid, and reactor walls, are at similar temperatures. Therefore, non-desired secondary reactions likely occur in the fluid phase, reducing the desired chemicals' production rate and process efficiency (Hamzehlouia et al., 2018). In the MW heating approach, however, heat is generated in the body of certain phases (commonly solid particles) owing to the high penetration depth of MWs and their interaction with dielectric material loaded in the reactors. MWs cannot directly heat the gas phase due to their negligible interactions with this phase (Hamzehlouia et al., 2018; Amini et al., 2019). Therefore, a considerable temperature gradient between the solid particles (either inert or catalyst) and fluid suppresses the fluid-phase side reactions, favors the production of desired chemicals, and likely leads to higher selectivity and conversion (Stefanidis et al., 2014; Hamzehlouia et al., 2018; Amini et al., 2019; Robinson et al., 2020). The rapid and selective features of MW heating also decrease the treatment time and energy loss during the process (Gupta and

Leong, 2007). The effects of MW irradiation on various multiphase (gas/liquid-solid phases) reaction systems are presented in Table 1.

Despite the applications of MW heating in multiphase systems, its effect on the kinetics of these reactions is still under debate. The main discrepancy is the presence of non-thermal effects, i.e., the interaction of MWs with materials at atomic/subatomic level and electron donation enhancement, in addition to the thermal effects, i.e., selective heating and formation of hotspots. In the present communication, we reviewed the impacts of MW heating on the reaction kinetics of multiphase reactions reported in the literature. We critically compared the experimental procedures in various references to propose the potential sources of discrepancy in the kinetic results.

Kinetics of gas/liquid-solid reactions

According to literature, MW heating has mainly thermal effects on reaction kinetics. For instance, hot spots generated

TABLE 2 kinetic studies of gas/liquid-solid systems under MW irradiations.

Process	Reactor ID (mm)/mass (g)	Temperature measurement technique	Changes compared to conventional heating		Rate controlling mechanism	Heating approach	Ref.
			E	K_0			
Methanol reforming	8 mm	Metallic thermocouple (top of the bed)	No change	No change	No change	Hybrid	Huang et al. (2016)
Thermal pyrolysis of bamboo sawdust	45 mm	Grounded metallic thermocouple (center of the bed)	↓ ^a	↓	NA	Hybrid	Dong and Xiong, (2014)
Char steam gasification	20 mm	Grounded metallic thermocouple (center of the bed)	↓	↓	No change (SCM ^c , VRM ^d)	Direct	Liu et al. (2019)
Iron production <i>via</i> hydrogen-reduction of Iron(II) sulfide –calcium oxide (FeS-CaO) mixture	35 mm	Radiation thermometer	↓	NA	•Conventional: reaction rate-limiting, and then diffusion rate limiting. •MW heating: gas diffusion rate-limiting	Direct	Amini et al. (2019)
Reduction of chromium ore fines containing coal	65 mm	Metallic thermocouple	↓	NA	No change	Direct	She-bin et al. (2008)
Carbothermic reduction of zinc oxide and zinc ferrite	NA	Metallic thermocouple on top of the sample	↓	NA	NA	Hybrid	Omran et al. (2020)
Pyrolysis of waste printed circuit boards	40 g sample in MW-TGA	Metallic thermocouple	↓	↓	Under MW heating, reaction order decreased	Hybrid	Sun et al. (2012)
Pyrolysis of lignocellulosic biomass	Quartz crucible (3–5 g)	Metallic thermocouple	↓	↓	NA	Hybrid	Luo et al. (2017)
Reduction of copper oxide (CuO)	Pelletized particles (8 × 2 mm)	IR ^e thermometer	↓	↓	NA	Direct	Fukushima et al. (2013)
Pyrolysis of wood sawdust for phenolic rich compounds	NA	IR thermometer	↓	NA	NA	Hybrid	Luo et al. (2017)
Paper cups pyrolysis	15	Metallic thermocouple	No change	No change	At a high heating rate, decomposition of cellulose was direct from feedstock to products rather than producing intermediates	Hybrid	Dominguez et al. (2007)
NaBH ₄ hydrolysis	15	NA	↓	↓	By increasing heating rate under MW irradiation, reaction order decreased	Direct	Yang et al. (2020)
Synthesis of phytosterol esters	200 ml	NA	↓	↑ ^b	NA	Direct	Perry et al. (2002)

^a: decreasing,

^b: increasing,

^c: shrinking core model,

^d: volume reaction model,

^e: infrared.

during the MW treatment of solid beds enhance the reaction rate as their temperature is significantly higher than the average bed (or bulk) temperature (Amini et al., 2019; Amini et al., 2021). The hot spots can form microcracks inside the solid materials and enhance internal mass transfer. For instance, MWs selectively heat some components of iron oxide minerals, generating a temperature gradient between different components within

an ore particle. Accordingly, micro-cracks are generated throughout the ore, enhancing gas diffusion into the ore, and improving the apparent kinetics of the reduction reaction (Amini et al., 2019).

In some discrepant cases, the non-thermal effect of MWs is also reported as a driving force in accelerating reactions (Hu et al., 2020). For instance, it was reported that a decrease in the activation energy (E in Eq. 1) during carbothermic reduction

of zinc oxide and zinc ferrite, which occurs at a lower temperature compared to conventional heating (Omran et al., 2020). In addition, Fukushima et al. (Fukushima et al., 2013) studied the reduction of copper oxide (CuO to Cu₂O) under MW heating. They reported that, compared to the conventional heating, E decreases to 2/3 and 1/3 under electric- and magnetic-field, respectively, due to the non-thermal effect of MWs. In contrast, others believe that decreasing the activation energy is due to underestimating hot spots temperature (Hamzehlouia et al., 2018; Amini et al., 2021). Accordingly, the measured temperature was the bulk temperature, while catalyst/solid particles had noticeably (50–300°C) higher temperatures owing to selective heating *via* MWs (Gupta and Leong, 2007; Farag and Chaouki, 2015; Hamzehlouia et al., 2018; Amini et al., 2021). Eq. 1 shows the general equation for reaction rate and its parameters.

$$R_i = k_0 \exp\left(\frac{-E}{RT}\right) f(C_i) \quad (1)$$

where R_i is the reaction rate of component i , k_0 is the pre-exponential factor, R is the universal gas constant, T is the absolute temperature, and C_i is the concentration of component i .

In the case of k_0 , both the increase (Yadav and V Borkar, 2006; Adnadjevic and Jovanovic, 2012) and decrease (She-bin et al., 2008; Dong and Xiong, 2014; Liu et al., 2019) have been reported under MW heating. However, k_0 is known as collision frequency that should not decrease under MW heating (Farag and Chaouki, 2015). In MW transparent fluids, no change is expected in k_0 compared to the conventional heating approach. The Effects of MW heating on the kinetic parameters of some thermal/catalytic reactions for gas/liquid-solid systems are presented in Table 2. Reactor diameter/sample amount, temperature measurement technique, and heating approach (direct: directly heating the sample *via* MWs, or hybrid: mixing the sample with MW receptor materials, such as silicon carbide (SiC) and activated carbon to heat the sample indirectly) are investigated in this paper.

Potential sources of discrepancy

Variations of reaction kinetic parameters in multiphase reactions under MW heating compared to conventional heating have potential reasons, such as reactor diameter/sample amount in a kinetic study (Benzennou et al., 2020a), heating approach (direct or hybrid) (Ren et al., 2022), temperature measurement (Gangurde et al., 2017; Ren et al., 2022), physical structure variation due to materials exposure to MWs (Amini et al., 2019), and interaction of MWs with polar molecules and free radicals (Wan et al., 2022). These sources are discussed below.

Effects of reactor diameter/sample amount and heating approach

The uniform temperature distribution in the reaction domain is essential to increase the accuracy of estimated kinetic parameters. Temperature non-uniformity in the bed is observed on both the macro-scale and micro-scale. The macro-scale temperature gradient of the bed is changed by variation of the sample's location in the cavity, the ratio of the waveguide to cavity impedances, and materials properties (affecting penetration depth). The main reason for the micro-scale temperature non-uniformity/hot spots is the difference in dielectric properties of materials in the bed (Ramirez et al., 2017; Amini et al., 2021).

Smaller reactor sizes and a few grams of solid mass are preferred to have a more uniform temperature distribution (see Figure 1), while effects of external and internal mass transfer limitations are eliminated/minimized either by increasing gas/liquid velocity or decreasing particle size, respectively (Benzennou et al., 2020a). To provide a uniform temperature distribution, we recommend applying a reactor diameter smaller than 10 mm (around 1/12 of MW wavelength at a frequency of 2.45 GHz), in fixed bed reactors adopted for intrinsic kinetic studies. In addition, employing fluidized bed reactors is another solution for an accurate kinetic study due to minimizing temperature non-uniformity (Farag and Chaouki, 2015; Samih and Chaouki, 2015). Impedance matching between the waveguide and cavity eliminates microwave reflection and minimizes the bed's macro-scale temperature gradient.

Heating of materials with weak MW interactions, e.g., plastics and biomass, in MW heating systems is challenging (Ren et al., 2022). Therefore, the hybrid heating approach is typically applied. However, due to variations in dielectric properties, it forms hot spots at MW receptors in the bed. For instance, pyrolysis of 40 g of waste printed circuit board in a MW-thermogravimetric analyzer resulted in both macro- and micro-scale hot spots during MW-assisted hybrid heating (Sun et al., 2012). In addition, MW-assisted pyrolysis of bamboo sawdust in a fixed bed reactor with a diameter of 45 mm (around 1/3 of the MW wavelength at 2.45 GHz) resulted in the formation of macro-scale hot spots (Dong and Xiong, 2014).

Effect of temperature measurement

Most MW reactors work based on temperature control mode, where the sample is heated to and kept at the desired temperature by coupling the feedback from an appropriate temperature measurement technique to the impedance tuner in the MW waveguide (Kappe, 2013; Gangurde et al., 2017). However, accurate temperature measurement of the gas/liquid and solid phases under MW heating is one of the critical issues in kinetic studies (Kappe, 2013; Gangurde et al., 2017). If the temperature is underestimated, the kinetic results show a fake enhancement.

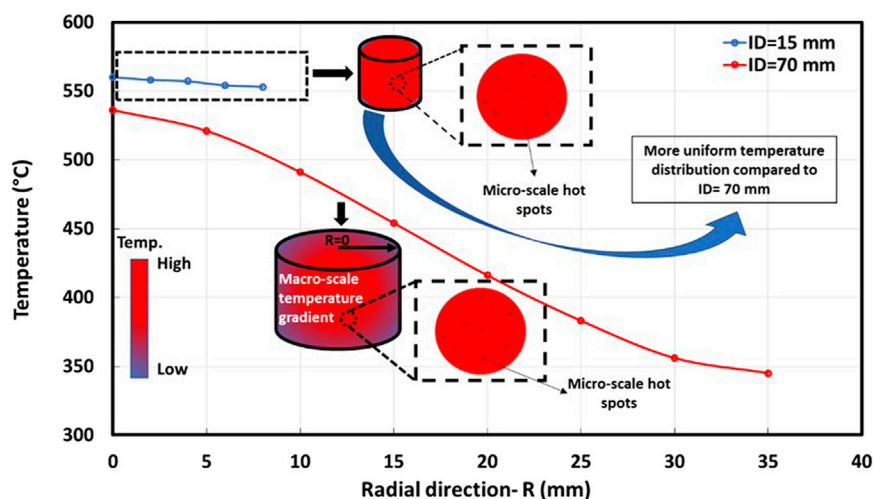


FIGURE 1

Effect of the internal reactor diameter (15 and 70 mm) on temperature distribution in a mixture of paper cups and graphite (adapted from ref. (Benzennou et al., 2020a)).

TABLE 3 Temperature measurement techniques in MW processing.

Technique	Main remarks	Ref.
Polymeric thermometer	<ul style="list-style-type: none"> • Limited for measuring the solid surface temperature • Inaccurate for gas-phase temperature measurement, especially during a reaction 	Gangurde et al. (2017)
Molecular thermometer	<ul style="list-style-type: none"> • Unsuitable for gas-solid reactions 	Gangurde et al. (2017)
Metallic thermocouple	<ul style="list-style-type: none"> • Interference with MWs • Spark formation 	Gangurde et al. (2017)
Fiber optic probes	<ul style="list-style-type: none"> • The difficulty in measuring local hot-spots temperature • More accurate than IR and metallic thermocouples • Immune to interference with MWs (depends on its design) • Limited temperature measurement range (0–300°C) • Fragile • Expensive 	Kappe, (2013)
IR-thermography	<ul style="list-style-type: none"> • Inaccurate due to the emissivity change during the reactions 	Gangurde et al. (2017)
Radiometry method	<ul style="list-style-type: none"> • Antenna made of planar printed steel that causes Interference with MWs 	Nguyen et al. (2020)
Air thermometer	<ul style="list-style-type: none"> • No interference with MWs • The difficulty in measuring local hot-spots temperature • Fragile 	Farag and Chaouki, (2015)

Currently, no technique can accurately measure the gas/liquid and solid local temperatures during MW heating (Nguyen et al., 2020). Various techniques, including grounded and shielded metallic thermocouples (Gangurde et al., 2017), fiber optic probes (Kappe, 2013), IR (Gangurde et al., 2017), polymeric (Gangurde et al., 2017), molecular (Gangurde et al., 2017), and air thermometers (Farag and Chaouki, 2015), as well as radiometry methods

(Nguyen et al., 2020), are employed in MW heated reactions. For kinetic studies, we need at least one contact sensor, e.g., thermocouple and fiber optic probe, or some other MW transparent sensors, to track the temperature changes during MW treatment of multiphase systems (Gangurde et al., 2017). Critical remarks regarding different temperature measurement techniques for MW heating systems are summarized in Table 3.

Effect of MWs interactions with polar molecules and free radicals

The activation energy is a characteristic of a chemical reaction implying the minimum required energy to initiate the reaction between reactants. Thus, a deep insight into the interactions (e.g., vibrations, disassociations, etc.) at an atomic scale under MW irradiation is essential to investigate their dependency on MWs. In liquid-solid reactions under MW irradiation, literature reported improvement in the kinetic. However, this improvement can be either due to hot spots formation in presence of solid/catalyst or dipolar/ionic molecules movement. For instance, Patil et al. (Patil et al., 2011) reported that the reaction rate constant for MW-assisted transesterification of oil on Barium oxide (BaO) catalyst is two order of magnitude higher than that for the conventional reaction. Although they provided no discussion/justification, it seems that they ignored the potential interaction between BaO catalyst and MWs that leads to a higher temperature at the catalyst surface compared to the bulk temperature (i.e., hot spot formation). BaO is a non-polar dielectric material with potential defects, including vacancies and interstitials, which couple with MWs to convert its energy into heat (Khujav et al., 2013). Khujav et al. (Ekinici et al., 2022) reported that the complete dissolution of BaO in hydrochloride acid under MW irradiation at 385 W takes less than 2.5 min, whereas it requires more than 20 min in conventional heating methods, indicating a significant interaction between MWs and BaO. Accordingly, a higher reaction rate during MW transesterification is attributed to the higher local temperature at the catalyst surface compared to the measured temperature, i.e., the bulk temperature, whereas the catalyst temperature is the same as the bulk temperature during conventional heating (Ekinici et al., 2022).

In liquid-solid MW heating-assisted reaction systems, MWs interactions can potentially affect ions and polar molecules. These interactions might result in a variation of kinetic parameters compared to conventional heating. For instance, the activation energy of Sodium tetrahydridoborate (NaBH_4) hydrolysis under MW heating (46.8 kJ/mol) is lower than that obtained under the conventional heating approach (66.9 kJ/mol) (Yang et al., 2020). The authors attributed this to the interaction between MW electric field and OH^- ions that accelerate hydrolysis, which was confirmed by a high-level pre-exponential factor (2.08×10^3 L/mol.min) under MW irradiation. In the study by Yang et al. (Benzennou et al., 2020b), accelerating ions under MW heating was also reported. However, the MW absorption capability of the solid/catalyst was ignored in these studies. If the solid

absorbs MWs, the improved kinetics in a liquid-solid system under MW irradiation is likely attributed to the formation of hot spots in the solid component, which provides a higher local temperature compared to the bulk. Moreover, the formation of intermediate products with MW interaction, which is locally heated, affected reaction kinetics by their rotation (Domínguez et al., 2007).

Unlike the gas phase, which is relatively transparent to MWs, liquid materials containing polar molecules or ions interact with MWs. Accordingly, a liquid ion's dipolar/rotational movement on a solid's surface can change the reaction pathway/mechanism (Yang et al., 2020). However, further theoretical/experimental investigations are needed to study the interaction of ions and polar intermediate with MWs in liquid-solid systems.

Future study

The development of new temperature measurement techniques, preferably non-intrusive ones, e.g., wireless thermometers, for local temperature measurement in MW systems is essential. In addition, the morphology variation of solid materials should be monitored under MW heating processes. Applying computational approaches like density functional theory (DFT), to investigate the effect of EM field on polar molecules in solid-liquid systems and consequently its impact on reaction kinetics/mechanism is an important concept that needs to be covered in future studies.

Conclusion

The effect of MW heating on the kinetic parameters of gas/liquid-solid reactions was reported in the literature. Underestimation and even sometimes overestimation of the reaction temperature in the reaction domain due to hot spots formation at micro and macro scales, and difficulty in measurement of these temperatures are the main sources of discrepancy in MW heating kinetic studies. Therefore, to the best of the authors' knowledge, there is no evidence in the literature confirming the presence of the non-thermal effect of MWs on the kinetics of gas-solid reactions, by now. Fixed bed reactors with a smaller diameter, e.g., 10 mm or operation under fluidized bed mode are suggested for kinetic studies to avoid noticeable temperature gradients within the reactor. However, changing the morphology of solid materials under MW heating can affect apparent kinetic parameters when the internal mass transfer controls the reaction rate. In liquid-solid systems with ions and polar molecules, electromagnetic fields likely accelerate the molecule's movement, which might

affect the reaction rate. The interaction of MWs with solid/catalyst should also be considered for potential hot spot formation to avoid an inaccurate reaction kinetics evaluation.

Author contributions

KA: Reviewed the literature, write the manuscript, revised the manuscript, discussed and concluded the findings AA: Reviewed the literature, write the manuscript, revised the manuscript, discussed and concluded the findings ML: Reviewed the literature, reviewed the manuscript, revised the manuscript, discussed and concluded the findings. JS: Reviewed the manuscript, revised the manuscript, discussed and concluded the findings. JC: Reviewed the manuscript, discussed and concluded the findings.

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Conflict of interest

Author JS was employed by the company Heirloom.

The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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