Verification of Microwave Effects on Molecular Clusters by Using Supersonic Molecular Jets

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Abstract: The origin of the specific effect of microwaves on chemical reactions (the microwave effect) was investigated by examining the effect of microwaves on small groups of molecules such as clusters. The origin of the effect was verified by introducing 2.45 GHz microwaves into a system equipped with a supersonic molecular jet and a special microwave feedthrough to record the fluorescence excitation spectrum of molecules. The carrier gas was bubbled through water and introduced into a phenol-filled sample holder to generate phenol-water clusters. Subsequently, it was confirmed that exposure of the phenol-water clusters contained in the molecular jet ejected from the pulse valve to microwave radiation increased the fluorescence derived from the phenol monomer. This is considered to occur because the phenol-water clusters in the molecular jet absorb microwaves and collapse, thereby increasing the abundance of phenol monomers. This result suggests that microwaves affect not only bulk systems but also small groups of molecules, and that local selective heating, which is one of the causes of the microwave effect, may occur.

Key words: electromagnetic wave effects, microwave chemistry, supersonic molecular jet, fluorescence excitation spectrum, microscopic heating

1 Introduction

Microwaves are electromagnetic waves in the approximate frequency range of 300–30 GHz. The discovery that microwaves can be used for heating was made in 1946 by Raytheon engineer P. L. Spencer during the development of radar microwave equipment\(^1\). Microwave energy has long been used to heat food; however, since the late 1980s, scientists began to consider the use of microwave energy as a heat source for chemical reactions. The first and well-known example of microwave chemistry was described in the report published by Giguère et al. in 1986\(^2\), after which microwaves were applied to many chemical reactions. In addition, the advantages of using microwaves over conventional heating using a heater or heat bath, such as a significant reduction in synthesis time and energy consumption, have been reported\(^3\)–\(^11\). Although microwaves have been widely applied in the field of chemistry, the use of microwaves in the synthesis of fatty acid methyl esters (FAMEs), which are used to produce biodiesel from animal/vegetable oils and waste oils, has increasingly received attention in recent years\(^12\)–\(^16\). The effect of accelerating the reaction by microwaves has been confirmed in the synthesis of FAMEs, and we are paying particular attention to the fact that the effect of microwaves has been exploited not only for heterogeneous catalyst systems\(^12\)–\(^16\) but also for homogeneous catalyst systems\(^14\)–\(^16\). According to our previous studies, a heterogeneous catalyst formed of components that easily absorb microwaves may be selectively heated to reach a higher temperature than the ambient environment to ultimately accelerate the catalytic reaction\(^17\). In the case of a solid catalyst, this phenomenon is presumably caused by a local high-temperature field formed in regions of the order of micrometers in which components with high microwave absorption characteristics are gathered. On the other hand, in a homogeneous catalyst system, the catalyst component is dissolved at the molecular level. As with the heterogeneous catalyst system, acceleration of the catalytic reaction in the homogeneous catalyst system as a result of selective heating of the catalyst indicates that selective heating by microwaves is a phenomenon that can occur at the molecular level.

As an example of selective heating with microwaves in a homogeneous system, Dudley et al. reported that acceleration of the Friedel-Crafts alkylation with the substrate of organic salts, designed to exhibit high microwave absorption properties, is faster under microwave heating condi-
tions than under conventional heating conditions and they conducted a detailed analysis of the reaction kinetics\(^{30}\). As a result, it was found that the reaction rate constant under microwave irradiation at 100°C was 7.8 times higher than that of the reaction driven by conventional heating at the same temperature. The reaction temperature was calculated by determining the rate constant of the reaction observed under microwave irradiation based on the Arrhenius equation, which corresponded to approximately 120°C. This result suggests that microwave selective heating may occur even in a microscopic region at the molecular level. When using microwaves for organic chemical reactions, including the FAME synthesis mentioned above, it is very important to understand the heating behavior in these molecular-level regions.

Usually, microwave heating envisions a bulk system composed of numerous molecules. When the system is irradiated with microwaves, molecules with a dipole moment seek to reorient according to an alternating electric field. However, the phase inversion of the microwave alternating electric field is extremely fast, and in bulk systems such as liquids and solids, the interaction between the molecules hinders the rotation of the dipole, preventing the reorientation of the molecules from keeping pace with the electric field, resulting in the input power being partially dissipated as heat\(^{30}\). Because heating proceeds by the above mechanism, heat dissipation cannot occur in isolated molecules as a matter of course, and it is conceivably necessary to form molecular groups such as clusters for heating to proceed. However, to the best of our knowledge, verification of the minimum size that would allow these clusters to dissipate heat in the microwave field has not yet been reported.

Therefore, in this study, we attempted to verify the effects of microwave irradiation on molecular clusters using supersonic molecular jet spectroscopy, which was devised by Kantrowitz et al. to create a high-density atomic beam\(^{30}\). Subsequently, Smalley et al. reported its application to the structural analysis of NO\(_2\) molecules\(^{21}\), and supersonic molecular jet spectroscopy has attracted attention as an effective technique for the structural analysis of polyatomic molecules. Molecular jet spectroscopy entails cooling the molecule to near absolute zero such that it is in the true ground state. Thus, the peak shape is significantly simplified compared to the broad and complex spectrum observed at room temperature, and an analyzable spectrum is obtained. The supersonic molecular jet technique has been used for the structural analysis of molecules in combination with various spectroscopic\(^{22, 23}\) and mass spectrometry\(^{24, 25}\) methods. In addition, molecular jet spectroscopy can be used to measure not only isolated single molecules, but also clusters formed by the association of multiple molecules.

Therefore, in this study, we used supersonic molecular jet spectroscopy to examine the behavior of water clusters, as a typical example of a substance that can be heated by microwaves. Initially, fluorescence excitation spectroscopy was used for structural analysis, but the water did not undergo fluorescence. Therefore, phenol, which is a fluorescent molecule, was used to evaluate the effect of microwaves by utilizing the fact that the spectral shape changes when water forms clusters with respect to phenol.

2 Experimental

A general schematic of the spectroscopic measurements using a supersonic molecular jet is shown in Fig. 1. A molecular flow is generated by ejecting a sample from a nozzle into a vacuum, together with helium as the carrier gas. At this time, because the velocity of the molecules exceed the speed of sound, it is referred to as a supersonic molecular jet. Immediately after ejection, the sample molecules and the carrier gas atoms present in excess around them collide with each other. This collision deprives the sample molecules of kinetic energy, and eventually the translation-temperature of the sample molecules is lowered such that the sample is cooled to an extremely low temperature, and the flow of the molecules is aligned in one direction. A schematic diagram of the experimental apparatus including the system that was used to record the fluorescence excitation spectra and microwave feed through is illustrated in Fig. 2. A sample holder filled with phenol and a solenoid-type pulse valve were installed on the upper surface of the vacuum chamber and connected to the equipment supplying the carrier gas. Selected experiments were carried out under the condition that a phenol-water cluster was stably generated as follows: the carrier gas was bubbled through the water, which was placed in a pressure-resistant con-
tainer immersed in a constant temperature bath at 20°C before being introduced into the sample holder. Excitation light was introduced through a window provided in the chamber in a direction orthogonal to the molecular jet ejected from the bulb. The excitation light was obtained by exciting a coumarin dye (coumarin 540-A) with a Nd: YAG laser and then converting the wavelength with a second harmonic generation (SHG) crystal to obtain a UV laser. The wavelength sweep range of the excitation light was arbitrarily set between 530 and 590 nm based on the wavelength of visible light before wavelength conversion, and the wavelength sweep speed was unified at 0.01 nm sec⁻¹. The UV laser wavelength (265–295 nm) after wavelength conversion was converted into a wave number corresponding to approximately 37736 to 33898 cm⁻¹. The fluorescence emitted by the excited phenol molecules was ampli-

Fig. 2 Experimental apparatus for fluorescence excitation spectrum measurement under microwave irradiation. Supersonic molecular jets are ejected perpendicular to the excitation light and receives microwaves emitted from an antenna placed directly under the molecular flow.

Fig. 3 Manufacturing drawing of microwave feedthrough (a) and photograph of feedthrough toward the inner surface of the chamber (b) and tip antenna (c). The explanation of the numbers in the manufacturing drawings is as follows; ① mount flange weld assembly, ② guide tube, ③ coaxial relay housing-1, ④ coaxial relay housing-2, ⑤ shaft brackets, ⑥ bracket, ⑦ coaxial cable, ⑧ type N coaxial weld sub assembly, ⑨ set collar.
fied by a photomultiplier tube (PM) and detected as an electrical signal. A semiconductor microwave oscillator (2.45 GHz, Fuji Electronic Industrial Co., Ltd.) was used as the microwave source, and custom-made microwave feed through equipment (ADCAP VACUUM TECHNOLOGY Co., Ltd.) was connected using a coaxial cable. The production drawing and appearance of the microwave feed through are depicted in Fig. 3. The tip has a disk antenna structure, from which microwaves are applied to the intersection of the molecular jet and excitation light directly above the disk. The antenna was designed using simulation software (Femtet software version 2019, Murata Software Co., Ltd.) to analyze the electromagnetic field and served the purpose of minimizing the reflected microwave power.

3 Results and Discussion
3.1 Verification of the effect of microwaves on phenol-(H₂O)_1-2 clusters

First, the fluorescence excitation spectrum of phenol was recorded without exposure to microwave radiation. The phenol used for the measurement was introduced into a sample holder and dried for 10 h while repeatedly opening and closing the pulse valve in a state where the vacuum chamber was depressurized to 10⁻⁶ torr. The measurements that were recorded by generating a molecular jet at a carrier gas pressure of 0.2 MPa are plotted in Fig. 4. The peak derived from the 0-0 transition of the phenol monomer was observed at 36346.9 cm⁻¹. In addition, characteristic peaks were observed at 35994.9 cm⁻¹ and 36257.3 cm⁻¹. These peaks were derived from the 0-0 transitions of phenol-(H₂O)_1 and phenol-(H₂O)_2, respectively. This indicated that a structure in which a small number of water molecules remaining in highly hygroscopic phenol formed a cluster with phenol can be observed with our apparatus. Next, to verify whether microwaves affect small clusters in which one or two water molecules are coordi-

![Fig. 4](image-url)  
**Fig. 4** Fluorescence excitation spectrum of phenol monomer and phenol-(H₂O)_1-2 clusters. The assignments were taken from refs [26-29].

![Fig. 5](image-url)  
**Fig. 5** The result of microwave irradiation for phenol monomer and phenol-(H₂O)_1-2 clusters. (a) total spectrum of fluorescence excitation, (b) comparison of the peak areas of phenol and phenol-(H₂O)_1-2 clusters in microwave ON and OFF cycle. The incident power of microwave was set to 65 W. The peak areas are normalized by the first microwave-OFF measurement.
nated to phenol, spectra recorded with and without microwave irradiation were compared. Although the measurement was repeated twice, the spectrum acquired in the presence of microwaves did not differ significantly from that acquired in the absence of microwaves across the entire wavenumber region (Fig. 5a). Therefore, the areas of the peaks of the phenol monomer and phenol-water cluster were compared (Fig. 5b). It was hypothesized that if local heating by microwaves were responsible for causing the collapse of clusters owing to the elimination of water molecules from phenol, the peak area derived from the clusters would decrease and the peaks derived from the monomers would increase, as shown in Scheme 1. However, the increasing/decreasing tendency of each peak changed randomly regardless of the presence or absence of microwave irradiation, and no significant difference could be detected. Therefore, the difference in peak area was judged to be within the measurement error. These results led us to conclude that microwaves do not affect the cluster size of small phenol-water clusters in which one or two water molecules are coordinated to phenol.

3.2 Verification of the effects of microwaves on large phenol-water clusters

Because the results suggested that microwaves do not affect small clusters with fewer than three molecules, we attempted to irradiate larger phenol-water clusters with microwaves. The results of the measurements we repeated five times every 20 min using undried phenol as the sample are shown in Fig. 6. In the first measurement, the phenol absorbed moisture, and because the phenol participated in clustering with many water molecules, the band structure became complicated. Specifically, the peak derived from the phenol monomer disappeared, and the peak broadened. Subsequently, it was confirmed that the phenol gradually dried as the measurement was repeated, and the intensities of the peaks attributed to the phenol monomer and phenol-(H₂O), (one molecule of phenol and water) increased. These observations revealed that the use of undried phenol can generate large phenol-water clusters. However, because the process whereby phenol gradually became drier was accompanied by a change in peak shape even if this change was the result of irradiation with microwaves, it is difficult to distinguish between drying and microwave irradiation. Therefore, it was decided to bubble the carrier gas through water and to combine water molecules equivalent to the vapor pressure with the sample to stably generate phenol-water clusters. This approach involving the use of wet carrier gas resulted in peak broadening and enabled us to confirm the coordination of many water molecules to phenol (Fig. 7). The broadened peak was stable over several measurements; hence, all subse-

![Scheme 1](image-url)
quent experiments were conducted by bubbling the carrier gas through water. Based on the hypothesis that the peaks derived from the phenol monomer intensify owing to the collapse of the cluster as microwave heating progresses, measurements focusing on the behavior of the phenol monomer were carried out. The wavelength of the excitation light was set to the excitation wavelength of the phenol monomer $\lambda_{275.05 \text{ nm}} = 36346.9 \text{ cm}^{-1}$, and the microwave radiation was turned ON/OFF 10 times every 30 s. This enabled us to compare the average peak intensity (Fig. 8), which was slightly higher for irradiation with microwaves, but the difference was as small as 0.74 %. Therefore, to obtain a larger difference, we examined the effect of the carrier gas pressure with the view of optimization. The higher the pressure of the carrier gas, the higher the collision frequency between the carrier gas atoms and the sample molecules becomes, and the higher the cooling efficiency. Then, because the cluster can be cooled immediately after ejection before collapsing, the probability of generating a larger cluster would be expected to increase. The pressure was varied between 0.2 and 1.1 MPa, and the change in the peak intensity of the phenol monomer as a result of switching the microwaves ON and OFF is compared in Fig. 9. The ON/OFF test was conducted 10 times at each pressure, and confirmed that microwave irradiation caused a significant difference at 0.2, 0.8, and 1 MPa (Table 1). The increase in the peak intensity of the monomer owing to microwave irradiation was most prominently confirmed at 0.8 MPa. Based on the heating mechanism of microwaves, the influence of microwaves is considered to be more likely to manifest itself when a larger number of water molecules form clusters, and the largest cluster was considered to have formed at 0.8 MPa. In contrast, above 0.8 MPa, the difference in peak intensity tends to decrease. This phenomenon is considered to have occurred because the relative amount of phenol and water decreased owing to the increase in the molecular ratio of the carrier gas, and conversely, it became difficult to form large clusters. Although the difference in peak intensity with and without microwaves at 0.8 MPa was still small at 1.1 %, subsequent experiments to evaluate the effect of microwaves were carried out by setting the carrier gas pressure to 0.8 MPa. The fixed wavelength measurement was replaced by the wavelength sweep measurement, and the effect of the microwave irradiation on the peak area of the phenol monomer-derived peak was evaluated. The results are shown in Fig. 10. Three repetitions of the microwave ON and OFF cycle confirmed that the area of the phenol monomer peak increased. On average, the monomer peak area increased
by 28% during microwave irradiation, showing the remarkable difference resulting from microwave irradiation. At this stage, the way in which large phenol-water clusters are formed under these experimental conditions remains unclear, and it would be necessary to use mass spectrometry to identify the minimum size a cluster would require to be affected by microwaves. Nonetheless, the results of this experiment suggest that microwaves may affect the cluster structure, at least within the n > 3 (more than three water molecules) region of phenol-(H₂O)ₙ.

4 Conclusion
The use of microwaves to irradiate phenol-water clusters in a molecular jet increased the areas of peaks derived from the phenol monomer. The peak area was assumed to increase because of the collapse of the cluster as a result of absorbing microwaves and dissipating heat. The results of this experiment therefore suggest the formation of local high-temperature fields within the microscopic region of the chemical reaction system under microwave irradiation. This phenomenon could not be experimentally observed, yet the result is considered to be a step closer to elucidating the microwave effect. In the future, we expect to identify molecular species capable of forming a high-temperature field in the chemical reaction solution by using molecular jet spectroscopy to conduct a systematic verification of the substrates and catalysts previously used in chemical synthesis reactions that have been confirmed to be accelerated by microwaves. This may further contribute to the elucidation of the mechanism of the microwave effect.

Table 1  Evaluation of significant differences by two-sided t-test of microwave on/off experimental results under each pressure condition. The t-test was performed with a significance level of 5% and degrees of freedom was set to 18.

| Pressure/MPa | 0.2 | 0.4 | 0.6 | 0.8 | 1.0 | 1.1 |
|-------------|-----|-----|-----|-----|-----|-----|
| t value     | 2.88| 0.48| 1.47| 2.61| 2.31| 1.20|
| p value     | 0.01| 0.64| 0.16| 0.02| 0.03| 0.25|
Contributions

D.S. conceived the study, designed the experiments and wrote the first draft. S.H. contributed to the writing of subsequent drafts and final draft of the article.

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Conflicts of Interest

The authors declare no conflict of interest.

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