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Development of a wideband microwave reactor with a coaxial cable structure

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HIGHLIGHTS

- A wideband microwave reactor with a coaxial cable structure was designed.
- Insertion of a truncated cone-shaped PTFE device was used as a method to reduce microwave reflection.
- The reflection ratio was less than 2% for a 0.1 M NaOH solution.
- Microwave heating at 915 MHz, 1.7 GHz and 2.45 GHz was accomplished.

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ABSTRACT

A wideband microwave reactor with an output that includes the 915 MHz and 2.45 GHz ISM (Industrial, Scientific and Medical) bands was designed and fabricated. The reactor structure incorporated a coaxial cable, and a liquid sample was placed in the space between the inner and outer conductors. Insertion of a truncated cone-shaped polytetrafluoroethylene (PTFE) device was used as a method to reduce microwave reflection over a wide frequency range. The reactor had a volume of 360 ml and was designed employing a 3D electromagnetic simulation. Ultrapure water and a 0.1 M NaOH solution were selected as the liquid samples and experimentally measured permittivity data for these liquids were employed during the reactor simulations. The measured reflection ratio exhibited the same trend as the simulation results between 800 MHz and 2.7 GHz. The reflection ratio was especially low in the case of the NaOH solution (less than 2%), although this value increased to more than 40% upon removal of the PTFE insert. Microwave heating tests demonstrated that this reactor was able to heat liquid samples at 915 MHz, 1.7 GHz and 2.45 GHz, with estimated microwave absorption efficiencies varying between 28% and 66% depending on the frequency, sample type and heating duration. The reflection ratio and heating data demonstrated that this reactor functioned over a wide frequency range between 800 MHz and 2.7 GHz. A non-uniform temperature distribution in the sample remained a challenge that must be addressed in future work.

1. Introduction

Microwave-assisted chemical reactions have attracted significant attention over many years, primarily because the microwave heating mechanism, so-called dielectric heating, is quite different from conventional heating. One feature of this process is internal heating; microwaves propagate deeply into materials and thus simultaneously generate heat on both internal and external regions of the substance. Another feature is selective heating; microwaves preferentially heat materials having a large dielectric loss. Together, these mechanisms allow shorter heating durations and highly efficient chemical reactions. Indeed, previous studies have reported that microwave synthesis can reduce reaction times [1] and that microwave heating is more efficient than convective heating [2].

With respect to the microwave apparatus, 2.45 GHz and 915 MHz frequencies are used in most cases [1,3–9], for both regulatory and economic reasons. Worldwide, these frequencies have been allocated for the purposes of microwave heating and are

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known as the ISM (Industrial, Scientific and Medical) bands. The prevalence of domestic microwave oven usage has led to a dramatic cost reduction in the case of high-power microwave units operating at 2.45 GHz.

The dielectric permittivity of a material is dependent on both frequency and temperature [10]. This implies that there may be an optimal frequency other than 2.45 GHz or 915 MHz that allows more effective microwave-assisted chemical reactions. On the laboratory scale, it is not necessary to use only the ISM bands due to the low levels of microwave power required to allow investigations of microwave-assisted reactions in small batches. However, there are currently no readily available units capable of generating microwaves in a number of frequency bands. In addition, the radiation type microwave reactors previously reported in the literature [6,9] are not suitable for wideband microwave irradiation because of the reactor frequency dependency that results from reflection, resonance and cutoff.

The objective of the present study was therefore to develop a wideband microwave reactor. For this purpose, we fabricated a coaxial cable structure, in which microwaves propagate in the transverse electromagnetic mode (TEM) with no cutoff frequency. In previous studies on microwave applicators [11] or radiators [12] using a coaxial cable, a liquid sample was irradiated with microwaves at the open end of the coaxial cable in the manner of dipole antenna-like radiation. In the present study, however, the microwaves were absorbed by a liquid sample based on propagation similar to that which occurs in a coaxial cable transmission line. This effect was obtained by positioning the liquid sample directly between the inner and outer conductors. In terms of the coaxial structure, a coaxial traveling microwave reactor (TMR) has been proposed to enable highly uniform microwave heating [13]. The TMR was designed via electromagnetic simulations only at 2.45 GHz; whereas the novelty of our designed reactor is realization of wideband microwave irradiation.

This wideband microwave reactor was designed using 3D electromagnetic simulations and had a target frequency range of 800 MHz to 2.7 GHz, a range that includes the ISM bands of 2.45 GHz and 915 MHz. Insertion of a truncated cone-shaped section of polytetrafluoroethylene (PTFE) device between the inner and outer conductors was used as a method to reduce microwave reflection over a wide frequency range. We verified the capabilities of the reactor, including the effectiveness of the PTFE insert, through electromagnetic simulations, microwave reflection measurements and microwave heating tests.

2. Materials and methods

2.1. Overview of the wideband microwave reactor

A cross-sectional schematic of the wideband microwave reactor along the axial direction is shown in Fig. 1. The reactor is rotationally symmetric along the central axis of the inner conductor. The outer and inner conductors produce the coaxial cable structure and a portion of a liquid sample is placed in the space between these conductors. A punched metal plate with 20 holes (each hole 6 mm in diameter) is mounted on the top surface of the liquid sample in order to prevent microwaves from leaking upwards through the sample and to avoid increases in the internal pressure of the reactor. An air release region above the plate buffers the inner pressure surge caused by sudden boiling. The inner and outer conductors, the metal plate and the wall of the air release area are all made of stainless steel (SUS 316L).

The microwave irradiation port consists of a commercially-available Type N connecter with a characteristic impedance of 50 Ω. A tapered section is inserted to smoothly connect the reactor and the microwave irradiation port. The characteristic impedance was maintained at 50 Ω by adjusting the ratio of the diameters of the inner and outer conductors. The space between the conductors was filled with PTFE to prevent the liquid samples from flowing out at the tapered section.

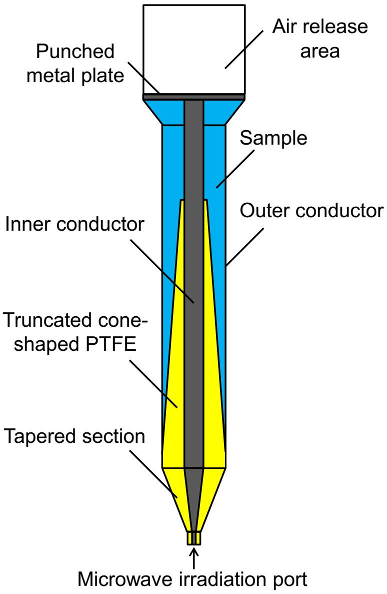

Fig. 1. A cross-sectional schematic of the wideband microwave reactor along the axial direction. The reactor is rotationally symmetric along the central axis of the inner conductor.

The truncated cone-shaped PTFE insert plays a role of reducing microwave reflection over a wide frequency range. In the present study, we used aqueous samples, as described in Section 2.2. Since the relative permittivity of such samples is usually high, smooth microwave propagation is essential to reduce microwave reflections at the boundary surface between the tapered section and the liquid sample. In Section 4.1, we discuss the effectiveness of the PTFE insert.

2.2. Liquid samples

Two liquid samples were selected: ultrapure water (henceforth water) as a dielectric sample and 0.1 M NaOH (henceforth NaOH solution) as a dielectric and conductive sample. The water was obtained from an ultrapure water system (ELGA PURELAB Flex3 PF3XXXXM1) and had an electrical resistivity greater than 18 MΩ cm. The NaOH solution was obtained by dissolving NaOH (JIS Special Grade, Wako Pure Chemical Industries, Ltd.) in the same water.

2.3. Permittivity measurements of samples

The permittivity of the liquid sample is an important factor to consider when designing a microwave reactor. In general, permittivity is dependent on temperature and frequency [10]. For the reactor to be useful, the microwaves must penetrate into the liquid sample with minimal reflections at all frequencies and temperatures.

Permittivity measurements of the liquid samples were conducted using the coaxial probe method [14], which is a common means of assessing the dielectric properties of liquids [15,16]. A diagram of the permittivity measurement system is shown in Fig. 2. The liquid sample was placed in a glass bottle and subsequently both heated and stirred using a hot plate stirrer (AS ONE