Thin-film Rotating Coaxial Reactor for Microwave-assisted Rapid Chemistry

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Abstract

This paper considers a prototype novel rotating reactor for microwave-assisted heating and chemistry. It consists of a coaxial waveguide with a dielectric hollow mixer turning around the length of the central conductor of this coaxial waveguide. A heating liquid flows in a narrow gap between the rotor and the outer conductor of the coaxial waveguide fed by microwaves. It is supposed that the acceleration of the conversation rate of chemical reactions is due to the excitation of Kolmogorov micro-vortices and the application of microwaves. This contribution studies theinitial results of microwave liquid heating, hydrodynamics, and mechanics in these reactors.

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Abstract. This paper considers a prototype novel rotating reactor for microwave-assisted heating and chemistry. It consists of a coaxial waveguide with a dielectric hollow mixer turning around the length of the central conductor of this coaxial waveguide. A heating liquid flows in a narrow gap between the rotor and the outer conductor of the coaxial waveguide fed by microwaves. It is supposed that the acceleration of the conversation rate of chemical reactions is due to the excitation of Kolmogorov micro-vortices and the application of microwaves. This contribution studies theinitial results of microwave liquid heating, hydrodynamics, and mechanics in these reactors.

Keywords: Microwave-assisted heating; rotating reactors; on-demand rapid chemistry hardware

1. Introduction

The development of green chemistry technologies supposes using processes and hardware allowing increased conversation rate, product selectivity and yields, better safety conditions, decreased energy usage, and product cost [1]-[3]. Some challenges are in the on-demand chemistry requiring the synthesis of products in small amounts at a low cost. Very often, the chemical hardware placed in laboratories and remote points should be controlled using IoT techniques without the presence of human personnel close to the hardware. For instance, a couple of satellites were launched into space with chemical equipment on the board.

Some of the promising hardware units are developed on the base of rotating reactors. Rotation is routinely used in liquid chemistry for ages to increase the rate of synthesis reaction and product yield [4],[5]. The coaxial designs or tube-in-tube reactors are distinguished by increased conversation rates of up to three orders. They are built on an inner rotating cylinder inserted into another stator tube. The heated mixture is pumped between the two mentioned cylinders. If the mixture's viscosity is relatively high and the gap is large enough, the Taylor-Couette vortices are generated along a cylindrical gap reactor. It allows better mixing and increases the conversation rate in orders [4]-[8].

Different designs were proposed with the liquid thin-film reactors[4],[7],[8]. In these units, a liquid mixture is pumped in a narrow sub-millimeter gap between a rotor and stator of cylindrical shape. It prevents the generation of large Taylor-Couette vortices, leading to some oscillation of product yield. Instead, the Kolmogorov sub-millimeter vortices are excited for fine mixing and rapid conversation of reagents into the prescribed products. In the mentioned reactors, the mixture is heated using electrical equipment outside of the reactors or in a vessel adjacent to the said tubing. Unfortunately, electrical heating requires increased temperatures of resistive wires that may lead to the firing of organic reagents in the case of a failure of reactor equipment.

Safer conditions are with microwave-assisted chemistry [9],[10]. It allows homogenous heating of polar liquids directly in reactors and increases the conversation rate. When exposed to microwave (MW) fields, liquids are often overheated over their boiling points, even at ordinary pressures. Further rising temperatures are allowed using pre-pressurised conditions in reactors. Such units are routinely used in research and educational labs and medium-volume industrial mini-plants.

Nevertheless, there are many attempts to develop new hardware for tailored chemistry. The MW reactors based on hollow cavities cannot be small because of the industrial frequency of 2.45 GHz and the employed cavity modes. Miniature items can be built on waveguides, allowing TEM (transversal electromagnetic) or quasi-TEM modes. They can be constructed of any small size [14], which is especially interesting in on-demand chemistry. Some are based on coaxial waveguides [11]-[23]. For instance, the radii of such reactors can be extended for volume increase using the multi-wire coaxial designs [12]. The residence time for high-loss polar liquids is improved using the dielectric-layered quasi-TEM waveguides [21],[22].

Coaxial designs are well suited for rotating liquid thin-film reactors. The first design of this kind was proposed in Ref. [23], where the central conductor is inserted inside a hollow dielectric rotor. The liquid under MW heating flows in the gap between this rotor and the outer coaxial conductor shield. It was supposed that the acceleration of conversation could be reached due to better mixing by exciting sub-millimeter Kolmogorov vortices in the narrow gap and volumetrical liquid heating by MWs along the whole reactor's length. This research aims to study the primary heating, hydrodynamic, and mechanical effects in a rotating structure similar to that proposed in Ref. [23].

2. Reactor Design Studied in Experiments

One of our preliminary designs is shown in Fig. 1. The left part of this figure shows a draft of the reactor consisting of a cylindrical stator of the length 60 mm, with the ID/OD adapted to RG-401 coaxial cable design. The hollow rotor is made of alumina (ID/OD= 2.6/4.67 mm) or quartz and connected to the motor's shaft using a flexible coupling. The golden-plated central conductor has a diameter 1.35 mm and a length of 48 mm. It is connected to a coaxial cable through the hermetic glass to a metal seal soldered to the stator.

The reactor can be matched to 50-Ohm, varying the batch height. Measurements and experiments are performed when the cold-ethanol-filled reactor shows the -10-dB-reflection coefficient registered by a vector network analyzer.

The liquid comes through the inlet to a miniature batch in the lower part of the reactor and farther to the gap between the rotor and stator (Fig.1B). The open design of this experimental device does not allow the pump rate over 1 mL/min to prevent liquid leakage through the open end. The fluid heated by MWs is evacuated through the outlet to an outer vessel. The liquid temperature is measured using a sensor installed inside the outlet, where a shallow level of MWs allows for thermocouple measurements [15],[16].

The reactor setup shown in Fig. 1B was connected to an MW generator and measurement units using hardware considered in Ref. [21]. It allows the incident and reflected powers to be registered using a reflectometer and two power meters. An MW indicator controlled parasitic irradiation from the reactor and connectors, and it did not exceed 0.05 mW/cm^2 for the applied MW power up to 40 W.

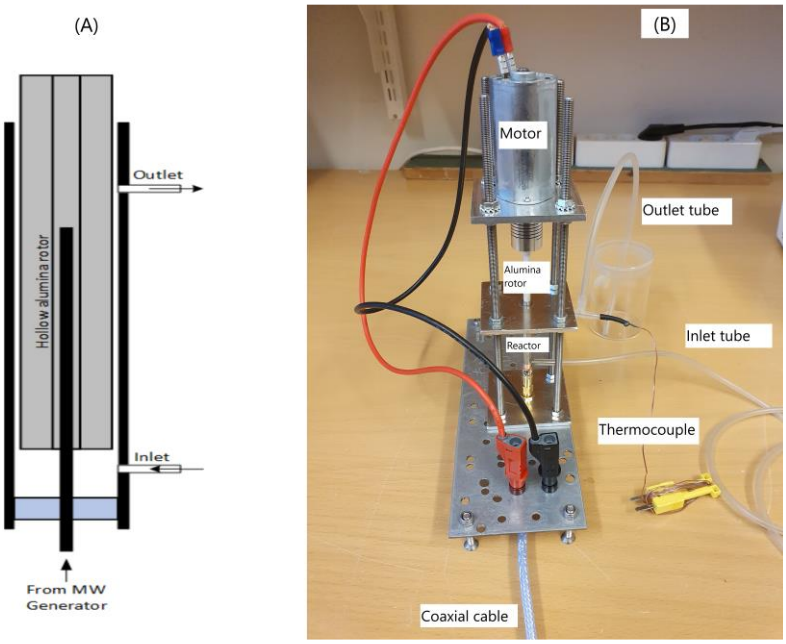

Fig. 1. Kinel 1.0 - A prototype of a thin liquid-film rotating MW coaxial open-end reactor. A)- Reactor draft; B)- Experimental reactor setup.

3. Results and Conclusions

Some results of the study of the manufactured prototype Kinel 1.0 are shown in Figs. 2-4. Two temperature trends are given in each figure for the static and rotating (4,000 rpm) regimes. A saturation of trends is seen at the temperature around $70^{\circ}\mathrm{C}$. It means that for all applied to the reactor, power values, the temperature inside the reactor reaches the boiling ethanol point $78.37^{\circ}\mathrm{C}$. The alcohol is cooled for several centigrades, moving from the heating zone to the outlet where a temperature sensor is installed. Rotation only slightly varies the temperature curves, but partly, this shaping depends on the oscillation of reactor input power caused by reflection from the complex reactor input impedance and non-ideal isolation of this generator from the reflected waves. The temperature trends do not show essential bubbling and severe variation of these curves in contradiction to coaxial reactors studied in Ref. [16]. It is caused by a submillimeter gap and mixing by the rotor.

In general, this prototype confirms what is proposed in Ref. [23], although the design needs further improvements through electromagnetic and thermal simulation using a numerical software tool. The liquid channel needs sealing to allow working at increased pumping rates. Avoiding oscillations of the input power from the MW generator requires additional implementation of a high-power ferrite isolator and better matching the reactor.

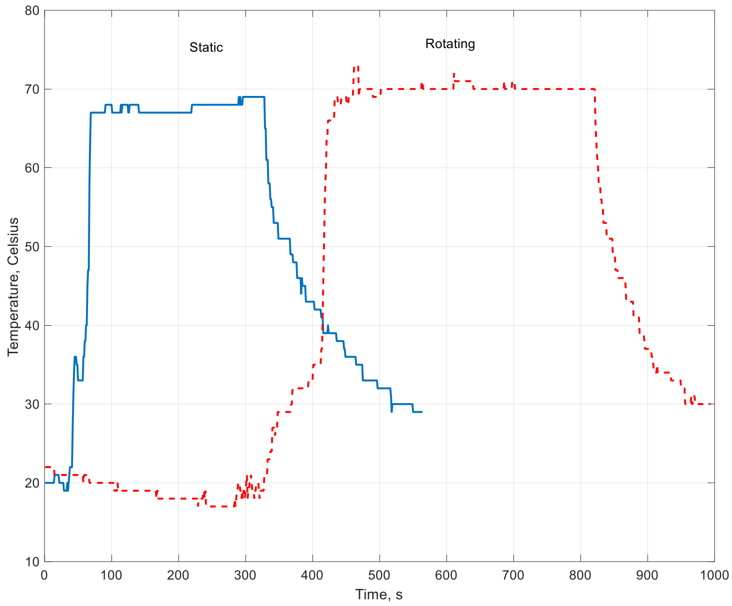

Fig. 2. Temperature trends for the reactor's static (S) and rotating (R) regimes given for 15.5/15.4 W average reactor input power. The absorbed average power values are: $P_{\mathrm{abs.}}^{(\mathrm{S})} \approx 6.5\ \mathrm{W}$ and $P_{\mathrm{abs.}}^{(\mathrm{R})} \approx 4.7\ \mathrm{W}$, correspondingly. Pump rate 1 mL/min.

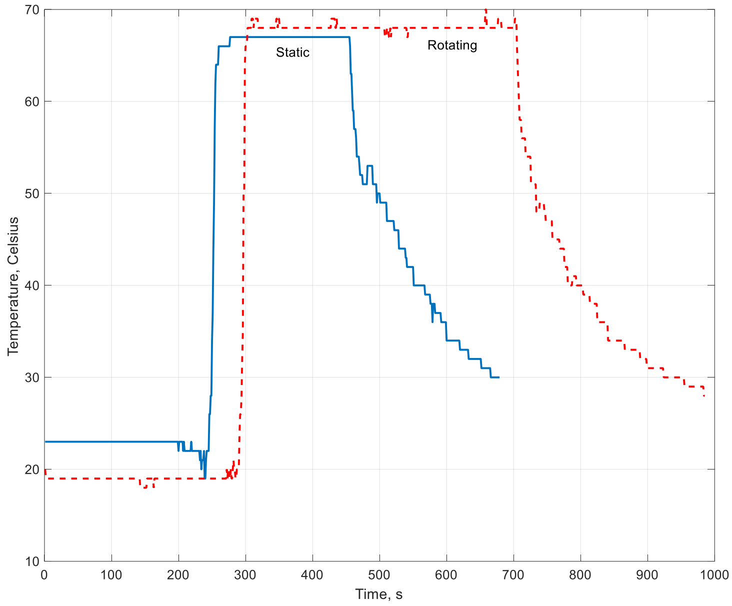

Fig. 3. Temperature trends for the reactor's static (S) and rotating (R) regimes given for 20.8/20.9 W average reactor input power. The absorbed average power values are: $P_{\text{abs.}}^{(\text{S})} \approx 9.8$ W and $P_{\text{abs.}}^{(\text{R})} \approx 10.7$ W, correspondingly. Pump rate 1 mL/min.

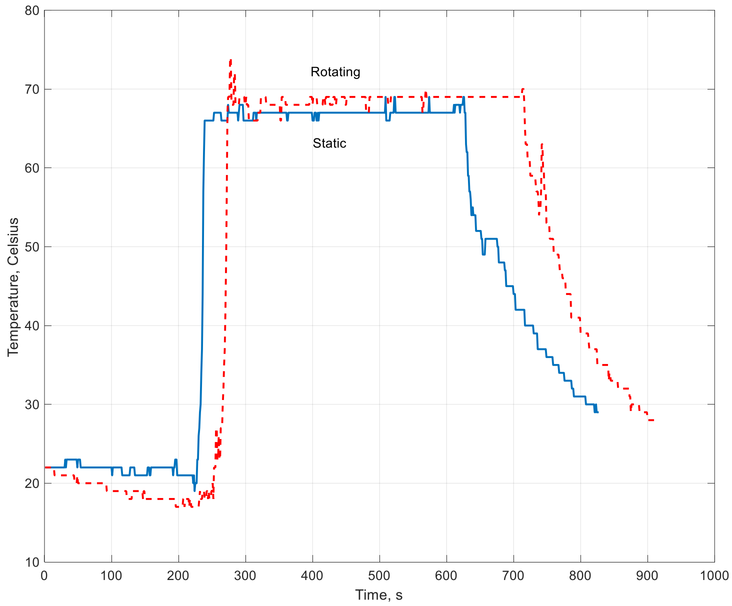

Fig. 4. Temperature trends for the reactor's static (S) and rotating (R) regimes given for 26.4/26.9 W average reactor input power. The absorbed average power values are: $P_{\text{abs.}}^{(\text{S})} \approx 15.1$ W and $P_{\text{abs.}}^{(\text{R})} \approx 14.4$ W, correspondingly. Pump rate 1 mL/min.

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