Development and Application of Microwave Loop Reactor Radiating through Leaky Coaxial Antenna(s)

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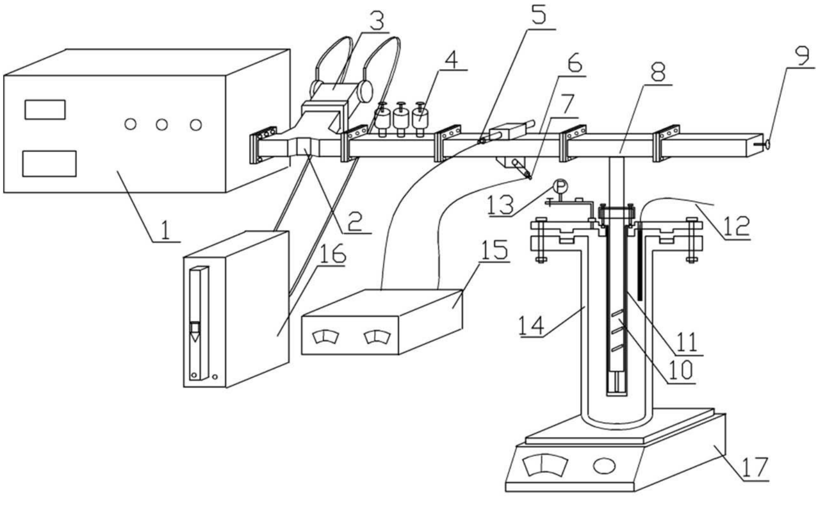


Figure 1. MW reactor: (1) MW power source, (2) three-port circulator, (3) water load, (4) three stub tuners, (5) input power detector, (6) directional coupler, (7) reflected power detector, (8) waveguide-coaxial converter (inside the waveguide), (9) terminal stub tuner, (10) leaky coaxial antenna, (11) sleeve, (12) temperature detector, (13) pressure meter, (14) reactor, (15) indicators, (16) water pump, (17) magnetic stirrer.

More details could be found in the below article:

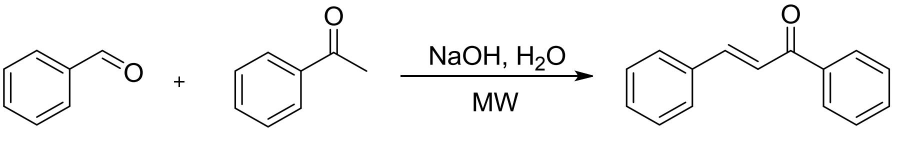
S. J. Lin, Y. Q. Peng, J. J. Hu, J. Y. Wang, Y. Li, G. H. Song. Development and application of a microwave reactor radiating through a leaky coaxial antenna. *Chem. Eng. Technol*. **2017**, 40(6), 1051-1058.

Figure 2. MW loop reactor: loop reactor (left); cover (middle); leaky coaxial antenna and PTFE sleeve (right). The cover is provided with three ports for connection of condenser, antenna insertion, and temperature detection (sampling) respectively. The antenna is centered in the reactor. The sleeve covers the exterior of the antenna to avoid the direct exposure of the antenna to the material.

Figure 3. Stainless steel shielding cover

The experiment was carried out in a glass reactor. During the experiments, a stainless steel shielding cover would be employed to minimize the MW leakage (almost not detected outside the cover). At the same time, the reaction process could be observed due to the small holes in the cover.

Description for all the experiments:



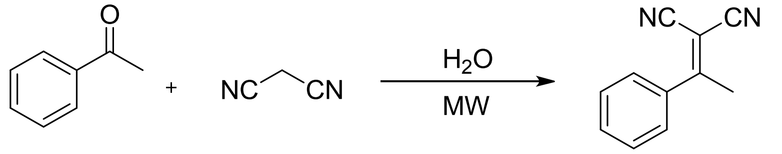
2.3 g (19.2 mmol) of acetophenone, 2.4 g (23 mmol) of benzaldehyde, 3.1 g (7.68 mmol) of NaOH and 14.4 mL water were charged into the commercial MW reactor (with a magnetic stirrer). It took about 0.5 min to heat the reaction mixture to reflux with the MW power of 300 W. The mixture was then stirred for 40 mins with an MW power of 70 W. After cooling down to the room temperature, 3.4 g of crude product was precipitated from the mixture. After recrystallization from ethanol, the desired chalcone was obtained with 80% yield (3.2 g).

28.8 g (0.24 mol) of acetophenone, 30.5 g (0.2875 mol) of benzaldehyde, 3.9 g (0.096 mol) of NaOH and 180.0 mL water were charged into the 0.3-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took about 1.5 mins to heat the reaction mixture to reflux with an MW power of 1,500 W. The mixture was then continuously refluxing and circulating for 40 mins with an MW power of 250 W. After cooling down to the room temperature, 48.0 g of crude product was precipitated from the mixture. After recrystallization from ethanol, the desired chalcone was obtained with 92% yield (45.9g).

57.6 g (0.48 mol) of acetophenone, 61.1 g (0.575 mol) of benzaldehyde, 7.7 g (0.192 mol) of NaOH and 360.0 mL water were charged into the 0.75-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took about 3 mins to heat the reaction mixture to reflux with an MW power of 1,500 W. The mixture was then continuously refluxing and circulating for 40 mins with an MW power of 450 W. After cooling down to the room temperature, 93.9 g of crude product was precipitated from the mixture. After recrystallization from ethanol, the desired chalcone was obtained with 90% yield (89.9 g).

115.2 g (0.96 mol) of acetophenone, 122.1 g (1.15 mol) of benzaldehyde, 15.4 g (0.384 mol) of NaOH and 720.0 mL water were charged into the 1.2-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took about 5 mins to heat the reaction mixture to reflux with an MW power of 1,500 W. The mixture was then continuously refluxing and circulating for 40 mins with an MW power of 750 W. After cooling down to the room temperature, 181.1 g of crude product was precipitated from the mixture. After recrystallization from ethanol, the desired chalcone was obtained with 87% yield (173.7 g).

230.4 g (1.92 mol) of acetophenone, 244.2 g (2.3 mol) of benzaldehyde, 30.8 g (0.768 mol) of NaOH and 1440.0 mL water were charged into the 2.4-L loop reactor radiating with two MW antennas (without a magnetic stirrer). It took about 5 mins to heat the reaction mixture to reflux with the MW power of 2×1,500 W. The mixture was then continuously refluxing and circulating for 40 mins with the MW power of 2×600 W. After cooling down to the room temperature, 355.1 g of crude product was precipitated from the mixture. After recrystallization from ethanol, the desired chalcone was obtained with 86% yield (343.5 g).



Amounts of 1.9 g (16 mmol) of acetophenone, 2.1 g (32 mmol) of malononitrile, and 12.8 mL water were charged into the commercial MW reactor (with a magnetic stirrer). It took about 0.5 min to heat the reaction mixture to reflux with the MW power of 300 W. The mixture was then stirred for 40 mins with an MW power of 75 W. After cooling down to the room temperature, 2.0 g of crude product was precipitated from the mixture. After recrystallization from 95% ethanol, the desired 2-(1phenylethylene) malononitrile was obtained with 70% yield (1.9 g).

Amounts of 24.0 g (0.2 mol) of acetophenone, 26.4 g (0.4 mol) of malononitrile, and 160.0 mL water were charged into the 0.3-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took approximately 1 min to heat the reaction mixture to reflux with an MW power of 1,500 W. The mixture was then continuously refluxing and circulating for 40 mins at the power of 220 W. After cooling down to the room temperature, 30.5 g of crude product was precipitated from the mixture. After recrystallization from 95% ethanol, the desired 2-(1-phenylethylene) malononitrile was obtained with 86% yield (28.9 g).

Amounts of 60.0 g (0.5 mol) of acetophenone, 66.0 g (1.0 mol) of malononitrile, and 400.0 mL water were charged into the 0.75-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took approximately 3 mins to heat the reaction mixture to reflux with an MW power of 1,500 W. The mixture was then continuously refluxing and circulating for 40 mins at the power of 450 W. After cooling down to the room temperature, 74.5 g of crude product was precipitated from the mixture. After recrystallization from 95% ethanol, the desired 2-(1-phenylethylene) malononitrile was obtained with 83% yield (69.8 g).

Amounts of 96.0 g (0.8 mol) of acetophenone, 105.6 g (1.6 mol) of malononitrile, and 640.0 mL water were charged into the 1.2-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took approximately 4.5 mins to heat the reaction mixture to reflux with an MW power of 1,500 W. The mixture was then continuously refluxing and circulating for 40 mins at the power of 700 W. After cooling down to the room temperature, 117.5 g of crude product was precipitated from the mixture. After recrystallization from 95% ethanol, the desired 2-(1-phenylethylene) malononitrile was obtained with 82% yield (110.2 g).

Amounts of 192.0 g (1.6 mol) of acetophenone, 211.2 g (3.2 mol) of malononitrile, and 1280.0 mL water were charged into the 2.4-L loop reactor radiating with one MW antenna (without a magnetic stirrer). It took approximately 4.5 mins to heat the reaction mixture to reflux with the MW power of 2×1,500 W. The mixture was then continuously refluxing and circulating for 40 mins at the power of 2×600 W. After cooling down to the room temperature, 230.2 g of crude product was precipitated from the mixture. After recrystallization from 95% ethanol, the desired 2-(1-phenylethylene) malononitrile was obtained with 81% yield (217.7 g).