

Supp info for

Microwave-assisted chemistry: A closer look at heating efficiency

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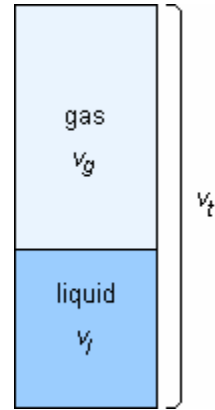
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1. Justification for neglecting the enthalpy of vaporization and gas heat capacity when calculating the change in internal energy

When considering a closed container of volume v_t that contains both liquid and gas with volumes V_l and V_g , respectively (see figure on the right), the corresponding moles in total as well as the liquid and gas phase can be denoted by n_t , n_l and n_g .

Since both the volume and the number of moles remain constant during heating, the following equations are valid:

$$\begin{aligned} V_t &= V_l + V_g \\ n_t &= n_g + n_l \end{aligned}$$



Furthermore it can be assumed that the ideal gas law is valid for pressures up to 20 bar (maximum operating pressure in the microwave synthesizers):

$$PV_g = n_g RT$$

where P is pressure, R is the gas constant and T the absolute temperature. Furthermore, the moles of liquid can be denoted as follows:

$$n_l = \frac{V_l \cdot \rho_l}{M}$$

where ρ_l and M are the liquid's density and molar mass, which combined with the previous three equations, gives:

$$n_g = \frac{P}{R \cdot T} \left(V_t - \frac{M(n_t - n_g)}{\rho_l} \right)$$

Which gives the following equation when solved for n_g :

$$n_g = \frac{P \cdot \rho_l}{R \cdot T \cdot \rho_l - P \cdot M} \left(V_t - \frac{M \cdot n_t}{\rho_l} \right)$$

If dn_g mole vaporizes in a time interval dt , the enthalpy changes with

$$\Delta_{vap} H \cdot dn_g$$

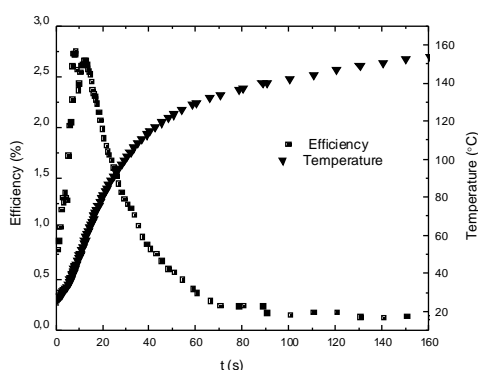
where $\Delta_{vap} H$ is the heat of vaporization of the liquid. When comparing this enthalpy change, in the range of the experiments carried out in this project, with values of $m_g c_g dT$ in the same time intervals dt , it follows that the vaporization energy is less than 0.5 % of the $m_l c_l dT$ value and, thus, may be neglected. Furthermore, it can be shown that the term $m_g c_g dT$ is also negligible.

2. Efficiency for microwave heating different volumes of demineralized water at small scale

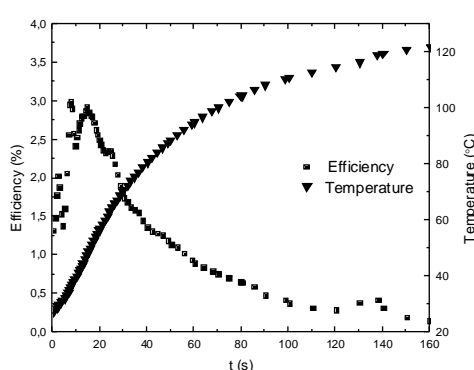
Table S1. Efficiency for microwave heating different volumes of demineralized water up to 100 °C with 300 W.

Sample volume	Average efficiency	Maximum efficiency
1 mL (0.5 – 2 mL vial)	2 %	3 %
2 mL (0.5 – 2 mL vial)	2 %	3 %
2 mL (2 – 5 mL vial)	8 %	16 %
3 mL (2 – 5 mL vial)	5 %	16 %
4 mL (2 – 5 mL vial)	13 %	23 %
5 mL (2 – 5 mL vial)	10 %	22 %

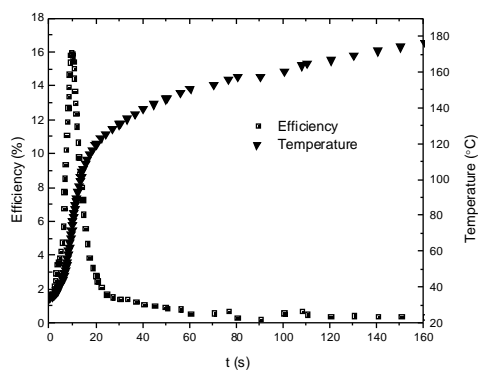
A: 1 mL in 0.5 – 2.0 mL vial



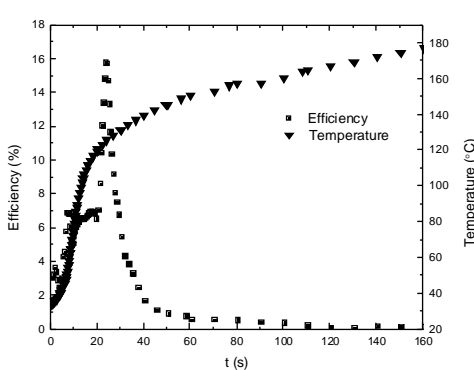
B: 2 mL in 0.5 – 2.0 mL vial



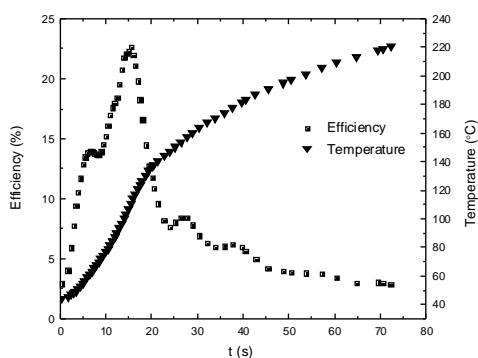
C: 2 mL in 2.0 – 5.0 mL vial



D: 3 mL in 2.0 – 5.0 mL vial



E: 4 mL in 2.0 – 5.0 mL vial



F: 5 mL in 2.0 – 5.0 mL vial

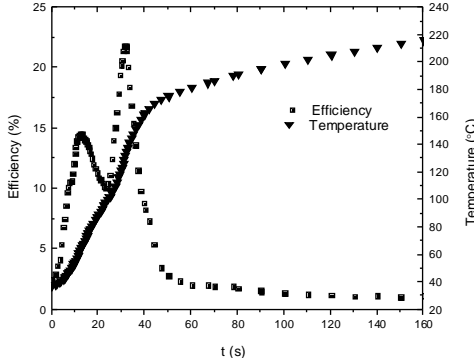


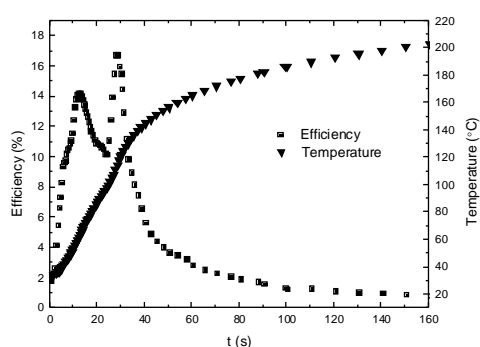
Figure S1. Temperature and efficiency profiles for microwave heating different small-scale volumes of demineralized water in 0.5 – 2.0 mL or 2.0 – 5.0 mL microwave vials with 300 W.

3. Efficiency for microwave heating different solvents at small scale

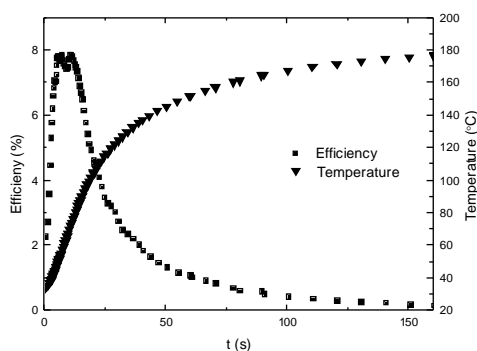
Table S2. Average heating efficiency for heating various solvents to 100 °C. Unless otherwise indicated, a volume of 5 mL is heated at 300 W.

Sample	Average efficiency	Maximum efficiency
Ultra pure demineralized water	10 %	17 %
Ethanol	6 %	8 %
Toluene	1 %	2 %
Diethyl ether	1 %	3 %
DMF (150 W)	11 %	17 %
1-butyl-3-methyl imidazolium chloride (BMIM-Cl; 150 W)	12 %	19 %

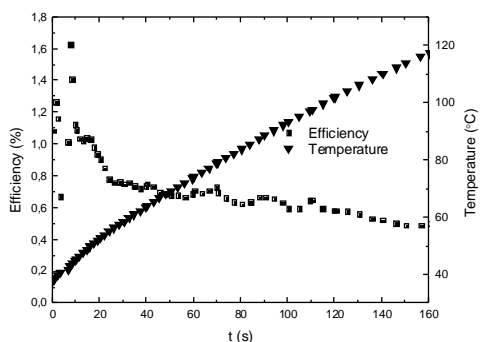
A: Ultra pure demineralized water



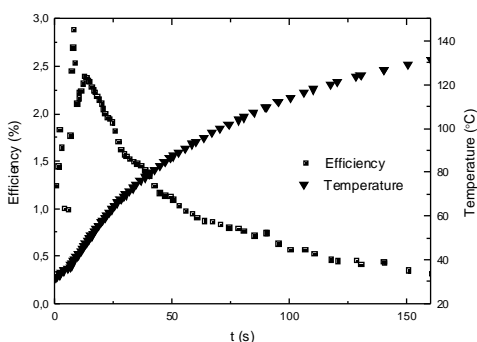
B: Ethanol



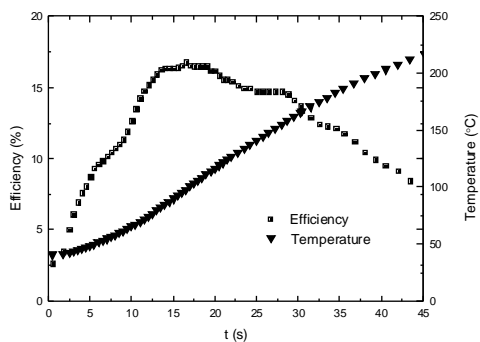
C: Toluene



D: Diethyl ether



E: *N,N*-dimethylformamide (150 W)



B: BMIM-Cl (150 W)

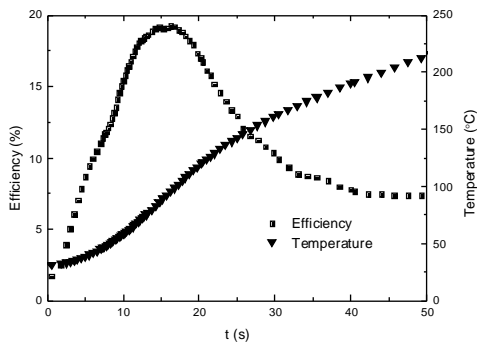


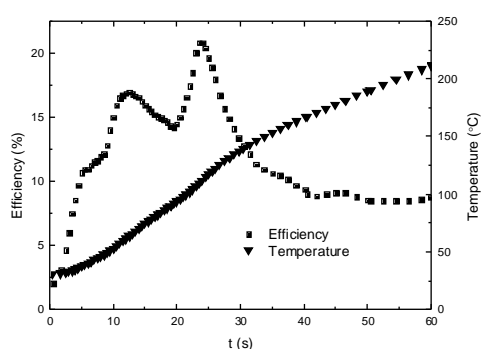
Figure S2. Temperature and efficiency profiles for microwave heating 5 mL of different solvents 2.0 – 5.0 mL microwave vials with 300 W or 150 W.

4. Efficiency for microwave heating aqueous NaCl solutions

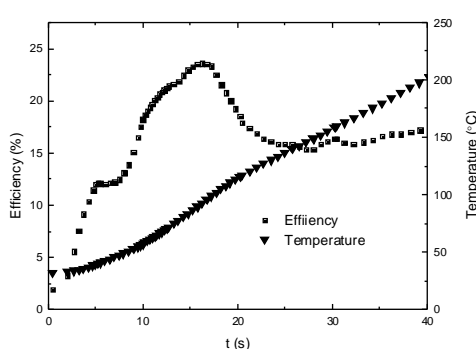
Table S3. Average heating efficiency for heating various aqueous NaCl solutions to 100 °C with 150 W or 300 W (volume of 5 mL).

Concentration NaCl (wt%)	Average efficiency 150 W	Maximum efficiency 150 W	Average efficiency 300 W	Maximum efficiency 300 W
0.03	14 %	21 %	12 %	21 %
0.10	14 %	21 %	15 %	24 %
0.60	21 %	32 %	17 %	28 %
1.80	22 %	33 %	17 %	30 %
5.0	21 %	32 %	17 %	27 %

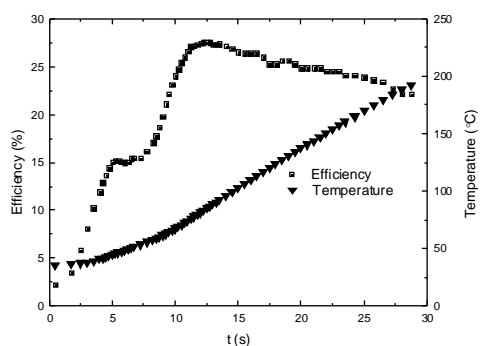
A: 0.03 wt% NaCl; 150 W



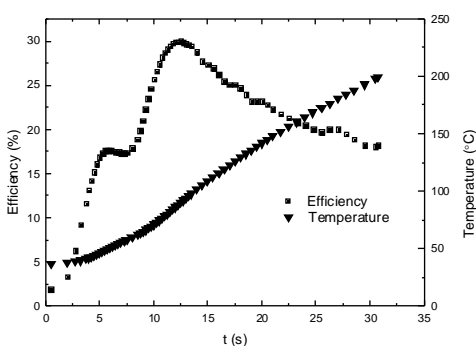
B: 0.10 wt% NaCl; 150 W



C: 0.60 wt% NaCl; 150 W



D: 1.80 wt% NaCl; 150 W



E: 5.0 wt% NaCl; 150 W

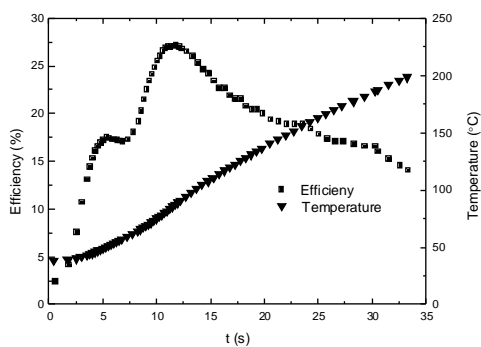
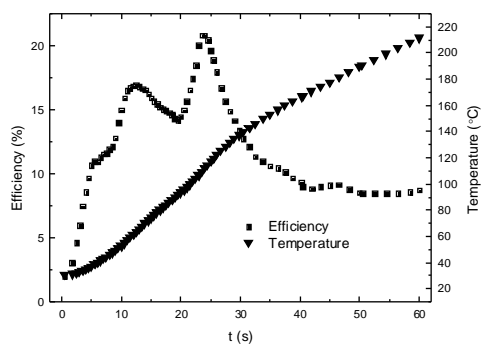
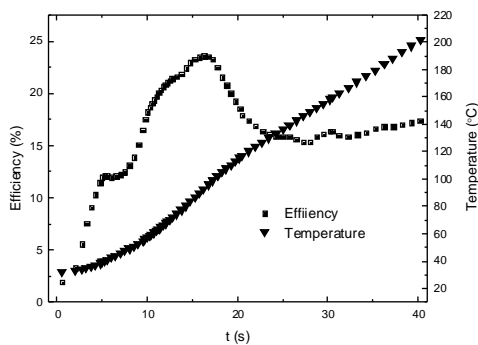


Figure S3. Temperature and efficiency profiles for microwave heating 5 mL of aqueous NaCl solutions in 2.0 – 5.0 mL microwave vials with 150 W.

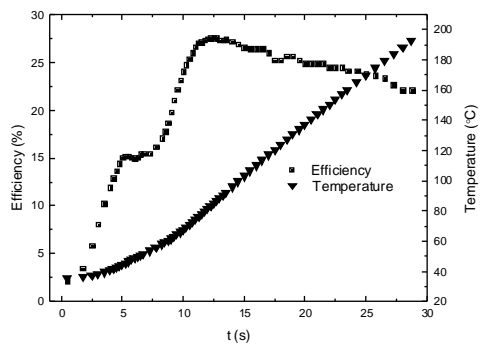
A: 0.03 wt% NaCl; 300 W



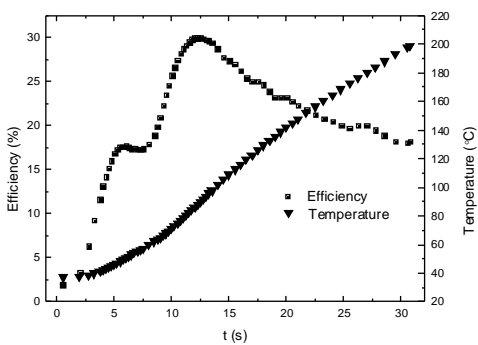
B: 0.10 wt% NaCl; 300 W



C: 0.60 wt% NaCl; 300 W



D: 1.80 wt% NaCl; 300 W



E: 5.0 wt% NaCl; 300 W

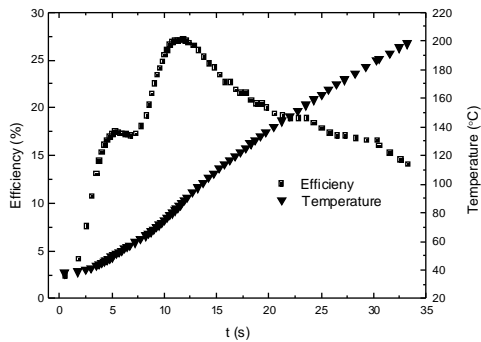


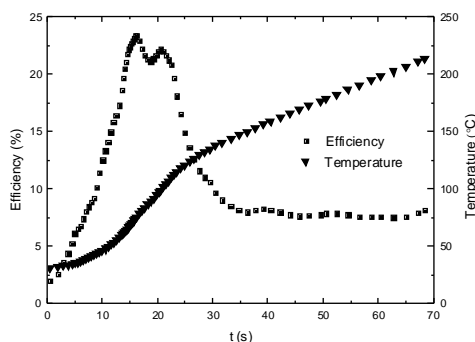
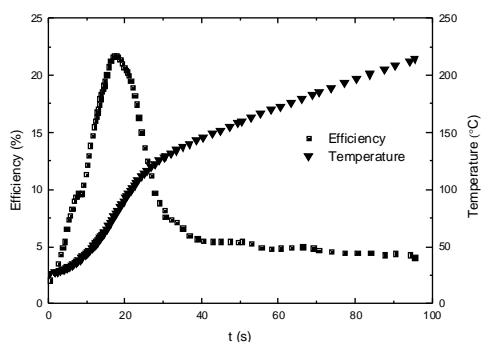
Figure S4. Temperature and efficiency profiles for microwave heating 5 mL of aqueous NaCl solutions in 2.0 – 5.0 mL microwave vials with 300 W.

5. Efficiency for microwave heating in the presence of SiC passive heating elements

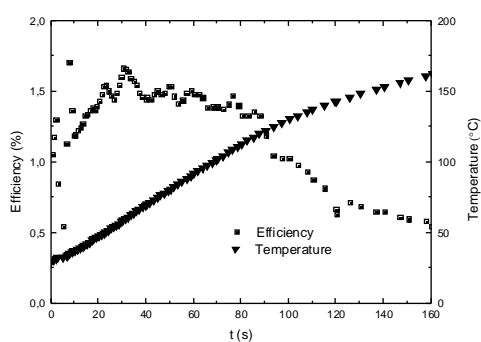
Table S4. Average heating efficiency for heating various solvents in the presence of passive heating elements to 100 °C with 150 W or 300 W (volume of 5 mL).

Sample	Average Efficiency		Maximum efficiency	
	1 element	2 elements	1 element	2 elements
Demineralized water (300 W)	1 %	13 %	22 %	23 %
Toluene (300 W)	1 %	6 %	2 %	10 %
DMF (150 W)	7 %	10 %	22 %	28 %

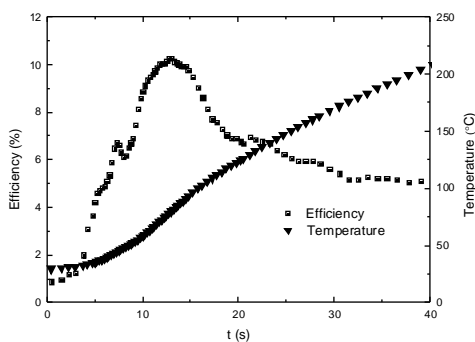
A: Demineralized water; 1 element; 300 W B: Demi-water; 2 elements; 300 W



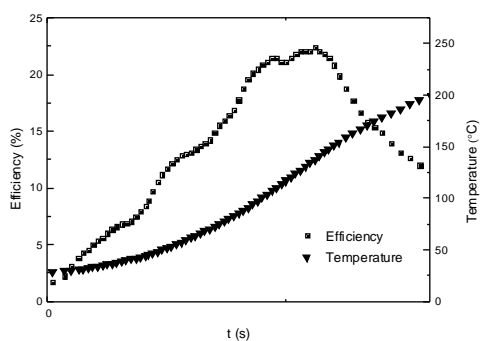
C: Toluene; 1 element; 300 W



D: Toluene; 2 elements; 300 W



E: DMF; 1 element; 150 W



F: DMF; 2 elements; 150 W

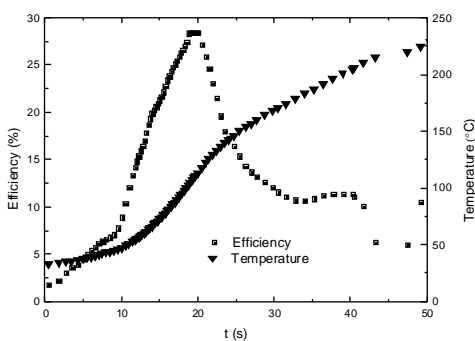


Figure S5. Temperature and efficiency profiles for microwave heating different solvents with 1 or 2 SiC passive heating elements.

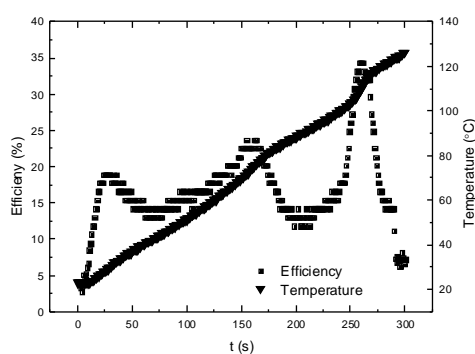
6. Efficiency for microwave heating of demineralized water at medium scale

Table S5. Average heating efficiency for heating demineralized water to 100 °C with various microwave power settings.

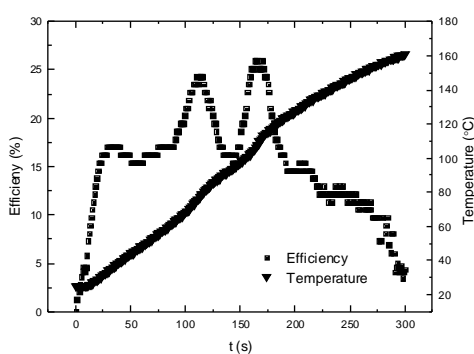
Power level	Average efficiency		Maximum efficiency	
	25 mL	50 mL	25 mL	50 mL
100 W	16 %	21* %	34 %	43 %
150 W	16 %	20 %	26 %	41 %
200 W	16 %	19 %	33 %	31 %
250 W	15 %	19 %	25 %	31 %
300 W	14 %	17 %	28 %	30 %

* 100 °C was not reached

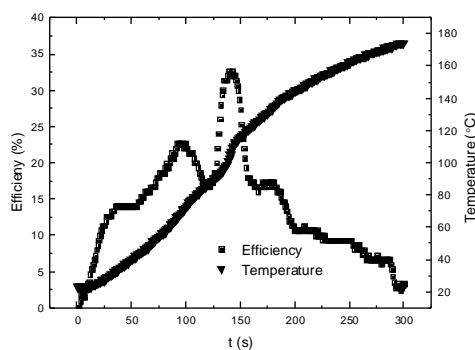
A: 25 mL; 100 W



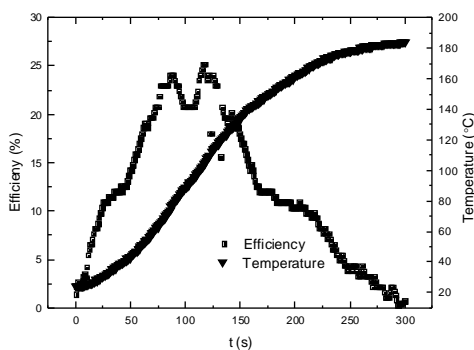
B: 25 mL; 150 W



C: 25 mL; 200 W



D: 25 mL; 250 W



E: 25 mL; 300 W

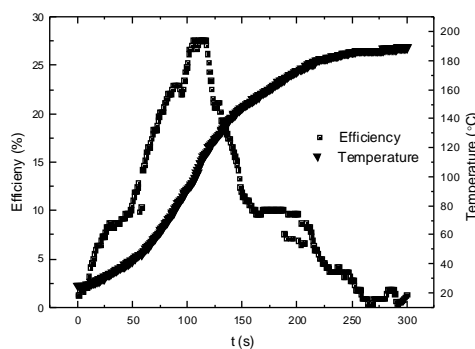
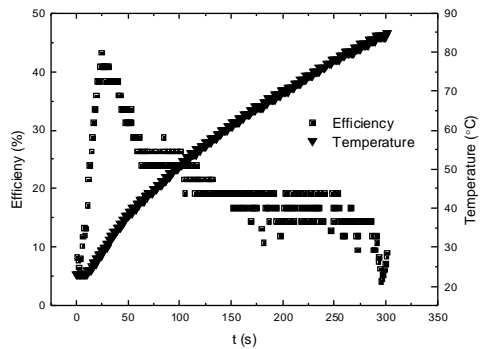
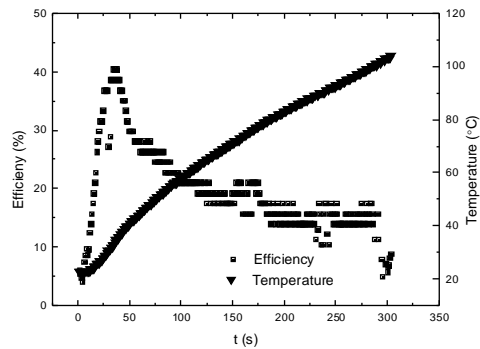


Figure S6. Temperature and efficiency profiles for microwave heating 25 mL demineralized water in with different power levels (CEM Discovery).

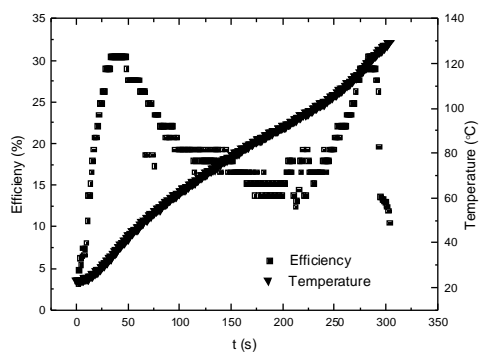
A: 50 mL; 100 W



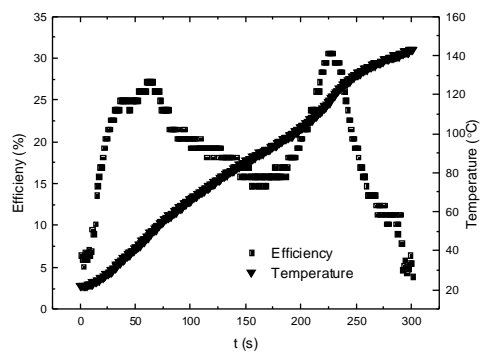
B: 50 mL; 150 W



C: 50 mL; 200 W



D: 50 mL; 250 W



C: 50 mL; 300 W

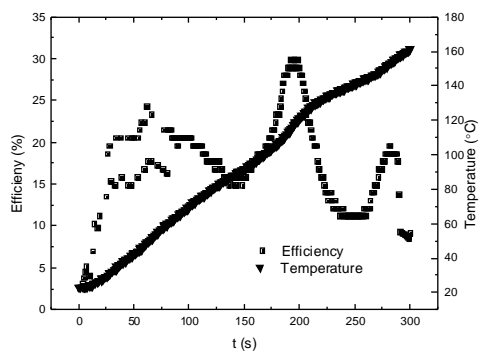


Figure S7. Temperature and efficiency profiles for microwave heating 50 mL demineralized water in with different power levels (CEM Discovery).

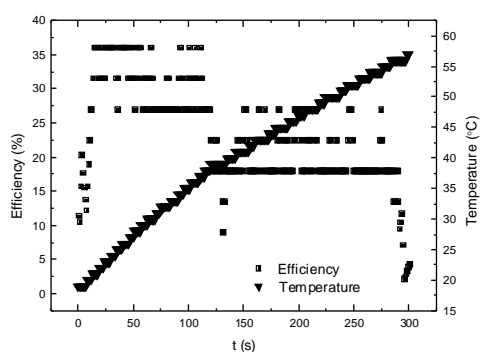
7. Efficiency for microwave heating of demineralized water at large scale

Table S6. Average heating efficiency for heating demineralized water to 100 °C with various microwave power settings.

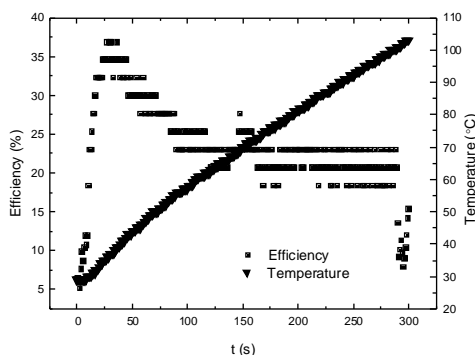
Power level	Average efficiency		Maximum efficiency	
	4 x 50 mL	8 x 50 mL	4 x 50 mL	8 x 50 mL
200 W	23* %	26* %	36 %	46 %
400 W	24 %	25* %	37 %	47 %
600 W	26 %	26* %	37 %	40 %
800 W	28 %	27 %	39 %	37 %
1000 W	30 %	31 %	37 %	39 %
1200 W	30 %	30 %	36 %	40 %

* 100 °C was not reached

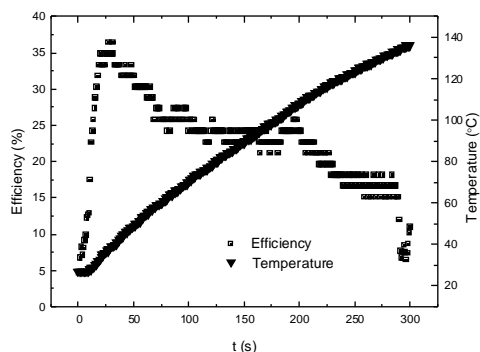
A: 4 × 50 mL; 200 W



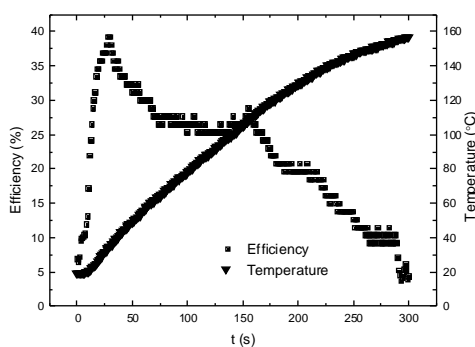
B: 4 × 50 mL; 400 W



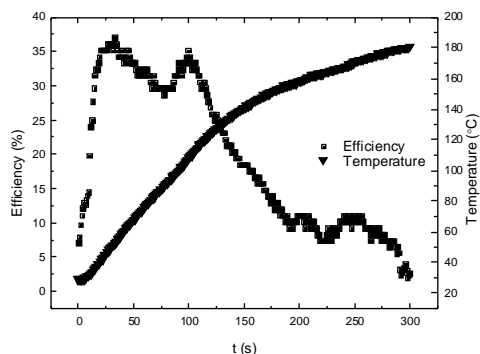
C: 4 × 50 mL; 600 W



D: 4 × 50 mL; 800 W



E: 4 × 50 mL; 1000 W



F: 4 × 50 mL; 1200 W

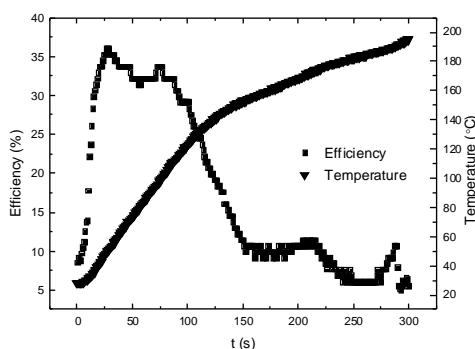
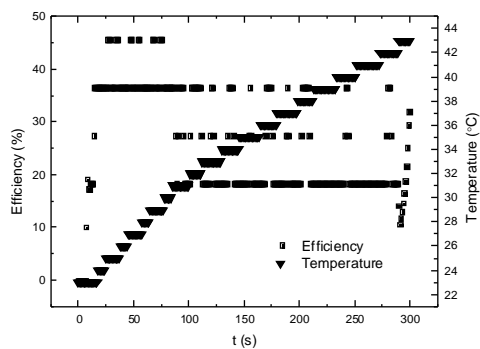
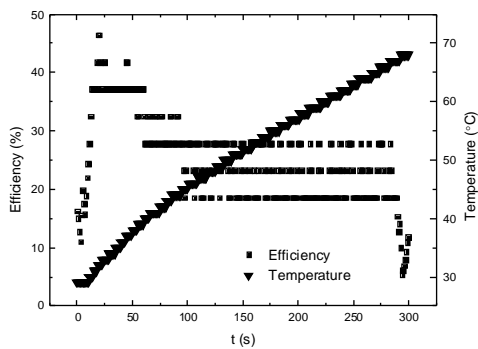


Figure S8. Temperature and efficiency profiles for microwave heating 4 × 50 mL demineralized water in with different power levels (Anton Paar Synthos3000).

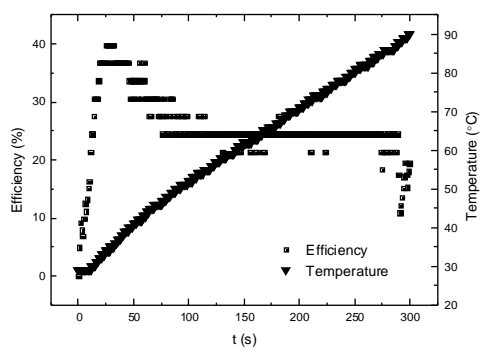
A: 8×50 mL; 200 W



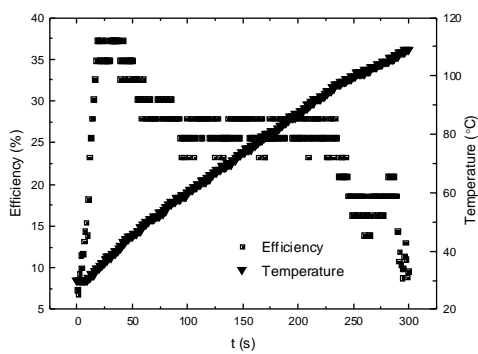
B: 8×50 mL; 400 W



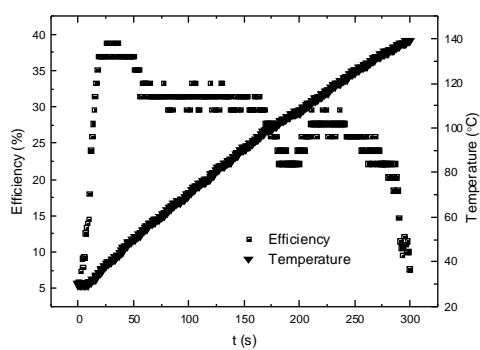
C: 8×50 mL; 600 W



D: 8×50 mL; 800 W



E: 8×50 mL; 1000 W



F: 8×50 mL; 1200 W

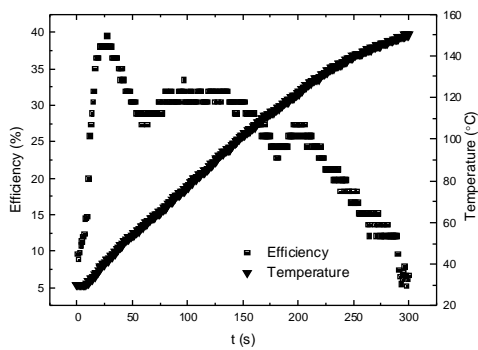


Figure S9. Temperature and efficiency profiles for microwave heating 8×50 mL demineralized water in with different power levels (Anton Paar Synthos3000).