Scale Up of Microwave Chemistry

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C-Tech History

1966  Electricity Council Research Center (ECRC) established as a publicly-funded research institute

1990  ECRC privatised along with the UK energy generation industry, becomes EA Technology

2000  Management buyout of EA Technology’s Energy Division, which becomes C-Tech Innovation
## Approach

- **Concept**: chemistry, physics, metallurgy
- **Pilot**: mechanical & electrical
- **Trials**: design & build
- **Production**: commissioning
Advanced Thermal Technologies

- Continuous Flow Chemistry
- Microwave Calcining
- Food and Drink Processing
- Continuous Cooking
- Advanced Induction Heating
- RF Curing and Moulding
- Plasma Surface Modification
C-Tech Innovation

Challenges of Scaling Up Microwave Chemistry

Microwave Chemistry Scale Up at C-Tech

Case Studies

Large Production Scale Microwave Chemistry
Challenges of Scale Up

Penetration Depth = Distance through the object being heated where the incident power drops to $1/e$ of that at the surface
Challenges of Scale Up

• Penetration depth and uniformity
• Materials
• Measurement and control
<table>
<thead>
<tr>
<th>Why Microwave Flow Chemistry</th>
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<tbody>
<tr>
<td>Faster</td>
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<tr>
<td>Faster reaction times</td>
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<td>Cleaner</td>
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<td>Rapid heating and absence of wall effects results in less side reactions</td>
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<tr>
<td>Greener</td>
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<td>Cleaner reactions means less purification/solvents/SMs. Energy saving MW flow vs batch</td>
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<td>Safer</td>
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<td>Less inventory of hazardous materials</td>
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Pilot Scale Reactor

- Flow rates up to 400 mL/min
- Dual feed vessels
- Pressurised receiver vessel
- Designed to process up to 20 L in a single run (can be operated for longer periods)
- Homogeneous reactions
- Light heterogeneous reactions
Equipment Specification

**Features**
Microwave power - 1-6 kW as standard (higher if required)
Temperature range - ambient to 250°C
Pressure - ambient to 30 bar
Flow rate - 5 mL to 1 L/min
Materials of construction - glass, fluoropolymer, stainless steel
Automatic temperature control

**Options**
Fibre optic temperature measurement
Halide resistant coating of steel parts
Complete plant or bare reactor
Case Studies

Reaction from Robinson Brothers

Equilibrium reaction - requires removal of aldehyde by-product to drive the reaction forward

Takes 4.5 days to produce 500kg, 30 batches per campaign

30 year old reaction
Case Studies

Reaction done in MW flow reactor
Temperatures between 120-150° C
Reaction times 2-10 mins
Gave >75% conversion to product and shows much less disulfide impurity than standard reaction
Difficulties in separating product from excess DMAPA used in the reaction
Currently investigating reduction of DMAPA eqvs and non aliphatic amines
• Difficult nucleophillic amination reaction – using conventional heating

• Reaction parameters quickly defined by MW chemistry - MW lab method gives double the yield with less solvent and readily available reagents

• Following day 18 L processed in 3.5 h to give 2.6 kg

• No method development required for scale up – saved an estimated 4 weeks in project time
Case Studies

- Thermal reaction
  - 92% conversion to product after 2h

- MW reaction
  - 295 mL/min (~1 min residence time)
  - Reaction went to 98% completion (analysis by GC/MS)
  - 4.08 kg/h of product

- Reaction run in pilot scale reactor in Sweden
  - Continuous operation — 32 hours
  - 140 L of reaction mixture giving 22 kg of isolated product
Case Studies

- Thermal reaction
  - 35% conversion after 8 h

- MW reaction
  - Reaction run at 75 mL/min (~4 min residence time)
  - 71% conversion to product
  - 307 g/h of product
Case Studies

• MW flow chemistry allows direct scale up from lab scale to plant scale without time consuming process development step
• MW flow chemistry allows rapid heating of solvents (sometimes above their boiling point)
  • Higher yields
  • Shorter reaction times
  • Less side products
Qulom MCP-6000

Temperature up to 250° C
Pressure up to 20 bar
Flow rates up to 200 mL/min
Dual feed vessels
Dual receiver vessels
Designed to process up to 10 L in a single run
(can be operated for longer periods)
Homogeneous reactions
Light heterogeneous reactions

Temperature up to 180° C
Pressure up to 20 bar
0.22L capacity
6kW
Qulom MCP-6000

20 bar, 215° C
20 mL capacity
1 kW, 2450 MHz
Hastelloy and quartz glass

ATEX rated
450 W solid state generator
@2450 MHz
20 bar
2 x 10 L pressurised receiver vessels
17 mL/min, 30 sec residence time
From 0 - 180° C in a single pass
Scale Up

• Scale up to 2000 kg/h throughput
• Maintain reaction temperature for 5.5 mins
• Requires 120 kW
• Designed as 4 separate modules with independent pumping, MW power supplies and MW power control
Note 1: To be confirmed the geometry of the holding pipe, and the number of static mixers
Note 2: Nitrogen will be supplied by the client, for the purging purpose
Scale Up
Scale Up
Scale Up

- Continuous flow microwave generator in 4 modular sections
- 5 x 6kW microwave generators @ 2450 MHz in each module section
- 20mm inside diameter quartz glass reactor tube with >1000mm heat application length
- Holding section with 5.5 minutes residence time at required flow rate
- Automatic control of outlet temperature, easy to use touch screen facility. Manual control option for microwave power also available, power can be set from 10% to 100%
- Working temperature up to 230°C
- Working pressure up to 10 bar
- Pressure relief and leak detection system
- Temperature and power analogue outputs
- Data logging of all process data of up to 50 data points with 20,000 entries each
Conclusions

Large scale microwave chemistry is possible

It's already being done at pilot scale

Continuous flow not batch

Materials and mechanical design are as important as microwave cavity design

Benefits

**Faster** reactions, more throughput

**Cleaner** - no hot oil, no fouling of hot surfaces

**Greener** - higher yields, less by-products, less catalyst, less waste

**Safer** - lower chemical inventories, easy temperature control

High throughput and continuous operation

Handles liquids and light slurries

Excellent chemical resistance