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EUROPEAN ROADMAP OF PROCESS INTENSIFICATION

- TECHNOLOGY REPORT -

<u>TECHNOLOGY:</u>
Microwave Equipment for Polymerization Reactions and Polymer Processing

<u>TECHNOLOGY CODE</u>: 3.3.3.4.3

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1. Technology

1.1 Description of technology / working principle

(Feel free to modify/extend the short technology description below)

Microwave (or dielectric) heating of materials has been known for a long time and development of microwave ovens has more than 50 years of history. The classical publication by Gedye et al. (1986) has opened a period of very intensive investigations of the microwaves effect on chemical reactions.
In general, microwave frequencies range from 0.3 to 300 GHz, which corresponds to the wavelength between approximately 1 mm and 1 m. Much part of this range is occupied by the radar and telecommunication applications and in order to avoid interference the industrial and domestic microwave appliances operate at several standard allocated frequencies, most often at 2.45 GHz. Molecules that have a permanent dipole moment (e.g. water) can rotate in a fast changing electric field of microwave radiation. Additionally, in substances where free ions or ionic species are present, the energy is also transferred by the ionic motion in an oscillating microwave field. As a result of both these mechanisms the substance is heated directly and almost evenly. Heating with microwaves is therefore fundamentally different from conventional heating by conduction. The magnitude of this effect depends on dielectric properties of the substance to be heated.
Authors generally agree about the ability of microwave heating to accelerate organic reactions and acceleration factors from several to more than thousand are reported. Also, increase of the product yield has been reported in some cases as a result of microwave heating.
The discussion about how the highly polarizing radiation of microwaves promotes and accelerates chemical reactions is still going on. Microscopic hot-spots, molecular agitation and improved transport properties of molecules have been mentioned as potential mechanisms of activation (Jacob et al., 1995). Other hypothetic effects, such as changing the molecules collision pattern, positioning of the transitions states or decrease of the activation energy in Arrhenius law were also postulated and discussed (Howarth and Lockwood, 2004). Recently more and more authors agree that the effect of microwaves has a purely thermal character (Lidström et al., 2001, Kuhnert, 2002, Kappe,. 2004), although for others this appears still a too simplistic explanation (Perreux and Loupy, 2001, 2002, De la Hoz et al., 2004).

1.2 Types and "versions"

(Describe the most important forms/versions of technology under consideration, including their characteristic features, differences and similarities)

Basically, any reaction including polymerization reaction can be carried out in two types of microwave reactors:

Monomode microwave reactor:
In the monomode devices a single standing microwave is created inside an electromagnetic waveguide. The sample is located in the place where the amplitude of the wave is the highest. In a basic modomode reactor the wave penetrates the sample cavity only from one side. Recently, CEM Corporation claims to have developed a laboratory microwave reactor, in which a cylindrical sample is uniformly irradiated with a monomode wave from several directions (Fig. 1).

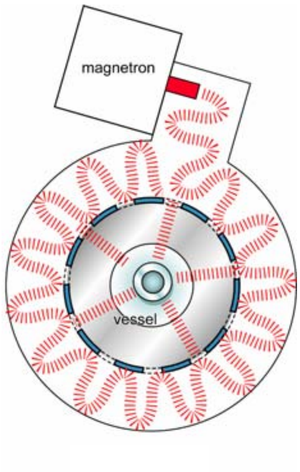

Fig. 1. CEM monomode microwave reactor

Multimode microwave reactor:

A multimode microwave reactor operates in a similar manner to a kitchen microwave oven. It contains a “field diffuser” to reflect the microwaves at different angles. Reflections of microwaves from the cavity walls results in different wave forms while standing waves are mostly avoided.

The achievable specific power density in monomode devices is usually greater than 1000 W/L and is up to ca. 2 orders of magnitude higher than in the multimode reactors. However, this high power density can be applied only to small volumes, as the maximum cavity size of the monomode reactors usually does not exceed 0.25 L, while multimode microwave devices cavities of hundreds of liters are often met. Accordingly, the potential application areas for the monomode and multimode microwave reactors differ considerably.

Other types of operations in polymer processing such as curing or pre-heating can be carried out in large industrial MW ovens (Fig. 2), such as those used for drying operations (See also TR 3.3.3.1).

Fig. 2. Industrial microwave oven for continuous drying operations

1.3 Potency for Process Intensification: possible benefits

(In Table 1 describe the most important documented and expected benefits offered by the technology under consideration, focusing primarily on energy; CO_2 emission and costs, providing quantitative data, wherever possible. Add other benefits, if needed).

Table 1: Documented and expected benefits resulting from technology application

Benefit	Magnitude	Remarks
Energy savings	No data are available for polymerization reactions, due to the lack of industrial-scale applications so far. For thermal processing of polymer parts a **7-fold reduction** in energy consumption has been reported polymer elements.	It can be assumed, that a successful application of MW in polycondensation processes could save between 30 and 60% of the energy, if current evaporation/condensation/incineration route was replaced by a membrane separation. At Henkel Surface Technologies in Cosne-sur-Loire plastic car components are produced via polymerization. The treatment requires heating of the workpieces. MW ovens have replaced the electric resistance ovens resulting in a 7-fold reduction of energy consumption.
Less CO_2 emission	No data available	
Cost savings	No industrial data so far, however can be substantial **(> 50%)** In curing of rubber a labor cost decrease by **30%** has been reported. Also a decrease of maintenance costs by **10-fold** has been reported	An unnamed US company producing expoxy resins decrease the number of shut-downs 4-fold and the maintenance costs 10-fold, by installing a MW pre-heater in their process. The affect was due to much less fouling/blockages occurring when the volumetric MW-heating is used.
Increased production capacity	**2-fold** increase reported	The above mentioned US company doubled its production capacity of an epoxy resin by applying a microwave pre-heater before the extrusion step. Much faster processing was enabled by the MW-heating.
Increased safety	No data available	Safety increase can result from: • Smaller inventory of chemicals in equipment • Possibility of instantaneous cut-off of the heat supply by switching off the microwave, in case of possible overheating/runaway conditions
Fouling prevention	See under Cost Savings	Contrary to the conventional heating by conduction, the volumetric MW heating creates no temperature gradients and uses no heat-exchange surfaces, which should lead to reduction of fouling.
Catalyst savings	**25%** reduction in catalyst consumption reported	The earlier mentioned US company decreased the catalyst consumption in an epoxy resin process from 0.8 to 0.6 kg/hr
Better product quality	**Substantial**; (new) polymeric products can be produced with properties not achievable with conventional heating.	Both, products with different Molecular Weight characteristics, different molecular structures and differente particle size distribution can be manufactured – see further under: "Potential applications discussed in literature"

1.4 Stage of development

Microwave reactors for polymerization reactions are still basically in the laboratory phase of development. Several vendors (see further) offer laboratory-scale microwave reactors, both monomode and multimode, batch as well as continuous.

Also, some attempts have been made to develop larger-scale continuous equipment for research purposes. An example of such continuous MW polymerization reactor, developed at Cracow University of Technology and based on the rotated quartz tube is shown in Fig. 3.

Fig. 3. Continuous MW polymerization reactor set-up at Cracow University of Technology

On the other hand industrial-scale microwave equipment is available for various operations in polymer processing, such as bonding/welding, curing or forming of plastic products.

2. Applications

2.1 Existing technology (currently used)

(Describe technology (-ies) that are conventionally used to perform the same or similar operations as the PI-technology under consideration)

Vast majority of polymerization reactions are carried out in batch or continuous stirred tank or tubular flow reactors with heating/cooling jackets and/or coils. Depending on process temperature various heat transfer media are applied, such as steam or mineral oils.

Other operations in polymer processing, such as bonding, curing or forming are conventionally carried out in electrically heated ovens.

2.2 Known commercial applications

(Is the technology broadly applied on commercial scale? In which process industry sectors is the technology most often applied: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? What is the estimated number of existing applications? In Table 2 provide the most prominent examples of realized applications and provide their short characteristics)

No commercial-scale applications of microwave reactors for polymerization reactions are known so far.

On the other hand industrial-scale microwave equipment has been applied on commercial scale for

Polymerization of plastic materials
Microwave heating can be advantageously used for temperature homogenization of initial mix of monomers and additives for polymerization.
It finds its use by the two main groups of polymers:
- thermoplastics, gained by simple polymerization
- thermosets, gained by polycondensation reaction.

In comparison with conventional heating, a substantial reduction of molecular weight of the plastic product can be reached by the microwave polymerisation of PMMA, PMA, PS (polymethylmetacrylate, polymethylacrylate and polystyrene) at comparable reaction conditions. Microwaves can be successfully applied to polymerize plastic material spread in a thin layer on paper, subsequently used in the manufacture of filters for car engines.

Bonding/welding of plastic parts
For bonding (gluing and welding) of plastics, microwave heating and particularly RF heating, one of the oldest and most successful applications, are used. Many lines are in operation, e.g. for welding of parts of casings for blood preservation for blood transfusion, and of other plastic materials.
The attained advances enable automatic control of the welding cycle also for processing of sophisticated, mass produced materials. An experiment - welding of thermoplastics by directed microwave energy - represents a novelty. The thermoplastics reinforced by long fibres cannot otherwise be welded by existing methods and must be glued. The Microwave welding raises the strength of samples up to three times in comparison with gluing and seems to be a suitable technology option.
Microwave heating is proved in two variants - bonding by direct heating of two polymer layers and other materials, or by using an interlayer.

Curing of rubber and epoxy resins
Probably the widest range of applications for microwave heating can be currently found in the rubber industry. The fact that most rubbed compounds will react to microwave energy enables the period of time required for heating to be considerably reduced. To achieve similar results by conventional heating methods the high temperature needed would cause degradation at the surface before the mass was fully heated. This is due to the poor thermal conductivity characteristics of rubber.
The heating of preforms (or blanks) prior to moulding whether on compression, transfer or ram injection presses has always been shown to be a worthwhile application. Ultimate cure and injection times can be reduced by up to 50%. As the power requirements for heating many of these preforms is relatively small, say 1 to 3 kW, the capital cost can generally be recovered in a short period, in some cases within 3 months.

Forming/shrinking of plastic products
By forming of plastic boards, microwave heating or pre-heating enables a homogeneous temperature in the whole volume of material, e.g. PVC, polystyrene and polypropylene. The commonly used thickness up to 6 mm can be processed in seconds. The main advantage of microwaves used for shrinking of plastic semi-products (plastic films, hoses etc.) is the exact control of the temperature. There is no standby power, and there is no heat inertia after switch-off; therefore no overheating occurs that would otherwise cause deterioration by partial baking, etc. This application can be used for the packaging and protecting layers technology.

However, a more detailed information about the companies and processes in which the technology is applied, is hardly available.

A quite recent interesting application of microwaves in polymer processing is the MW-assisted cracking of waste plastics to fuels (Global Resource Corp., USA).

Table 2. Industrial-scale applications of the Technology (existing and under realization)

Sector	**Company/Process (type)**	**Short characteristic of application**	**Production capacity /Plant size**	**Year of application**	**Reported effects**
Specialty chemicals & pharma	Major international company in Midwest (US)	Pre-heating of resin prior to extrusion	460 kg/hr	2001	• 2x more capacity • 2x shorter processing time; • 25% reduction of catalyst consumption • 10x less maintenance cost • Total cost saving of 865000 $/yr
Specialty chemicals & pharma	Henkel Surface Technologies	Heating of plastic car components	No data	2001	• 7x decrease in energy consumption

2.3 Known demonstration projects

(Are there any demonstration projects known related to the technology under consideration? In which process industry sectors are those projects carried out: large volume chemicals – specialty chemicals & pharma – consumer products – ingredients based on agro feedstocks? In Table 3 provide the short characteristics of those projects,)

No demonstration projects on microwave reactors for polymerization reactions and polymer processing are known.

Table 3. Demonstration projects related to the technology (existing and under realization)

Sector	**Who is carrying out the project**	**Short characteristic of application investigated**	**Aimed year of application**	**Reported effects**
				•

2.4 Potential applications discussed in literature

(Provide a short review)

The literature concerning the effects of the microwaves on ***polymerization reactions***, and polymer processing is rich. The reactions described in literature include:

- free radical polymerizations (e.g. methacrylates)
- controlled radical polymerizations
- emulsion polymerizations (e.g. polystyrene)
- ring-opening polymerizations (e.g. polyoxazolines)
- step-growth polymerizations
    - o synthesis of aramides, polyamides, polymides and polyimide-silica hybrid materials
    - o synthesis of polyesters and polyethers
    - o synthesis of epoxy resins
    - o synthesis of polyurethanes

In polymerization reactions microwave irradiation is able to produce a number of very interesting and commercially relevant effects.

Obviously, the most important effect of microwaves in those reactions is the **substantially increased speed of processing**. The MW-assisted reactions proceed much faster than by the conventional heating and the differences in reaction rates reach orders of magnitude. For instance Correa et al. (1998) observed ca. 70x acceleration of the emulsion polymerization of styrene.

Another important effect of the MW-heating in some polymerization reactions is synthesis of **polymers with different molecular weights than by the conventional heating**. The above cited Correa et al. (1998) observed the molecular weight of polystyrene prepared with microwaves to be 1.2 times higher than the one obtained with conventional method. In Fig. 4 a molecular weight vs. time curve for the direct polyesterification of butane-1,4-diol with succinic acid is shown (Velmathi, et al., 2005). One clearly sees that not only the MW-assisted process is much faster than the conventional but also in presence of the microwaves almost 2 times higher molecular weights are achieved than achievable with the conventional heating.

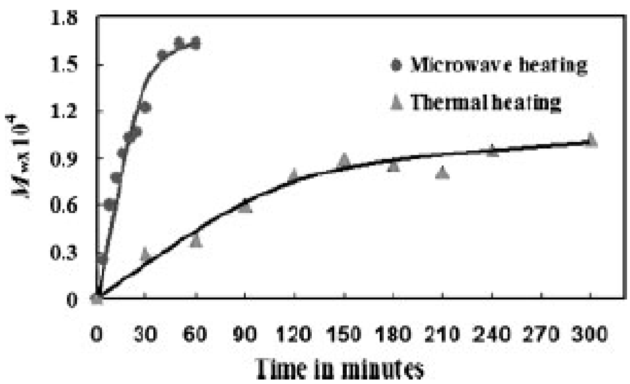

Fig. 4. Molecular weight vs. time curve for the direct polyesterification of butane-1,4-diol with succinic acid (Velmathi, et al., 2005).

In free-radical polymerizations microwaves were shown to substantially **increase the selectivity to the required product**. An example here is shown in Fig. 5, where the use of microwaves changed the selectivity in the synthesis of (methyl)acrylamides (Hoogenboom and Schubert, 2007).

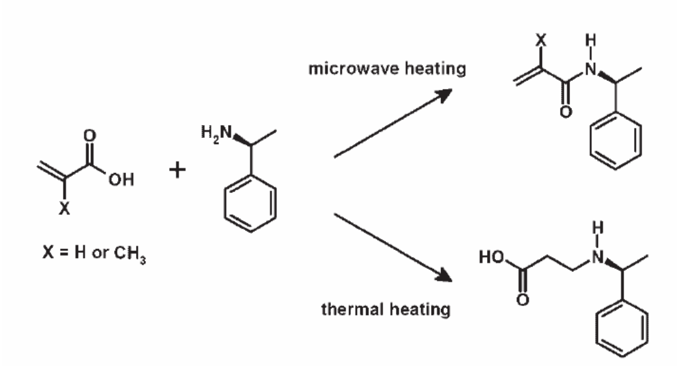

Fig. 5. Influence of MW-irradiation on the selectivity in the synthesis of (methyl)acrylamides (Hoogenboom and Schubert, 2007).

Finally, thanks to the MW-irradiation a different internal structure (e.g. porosity) of the product or a different particle size distribution (usually smaller and more uniform) can be obtained. Fig. 6 shows the comparison of the images of polystyrene particles obtained in emulsion polymerization with and without microwave irradiation (Xu, et al., 2005).

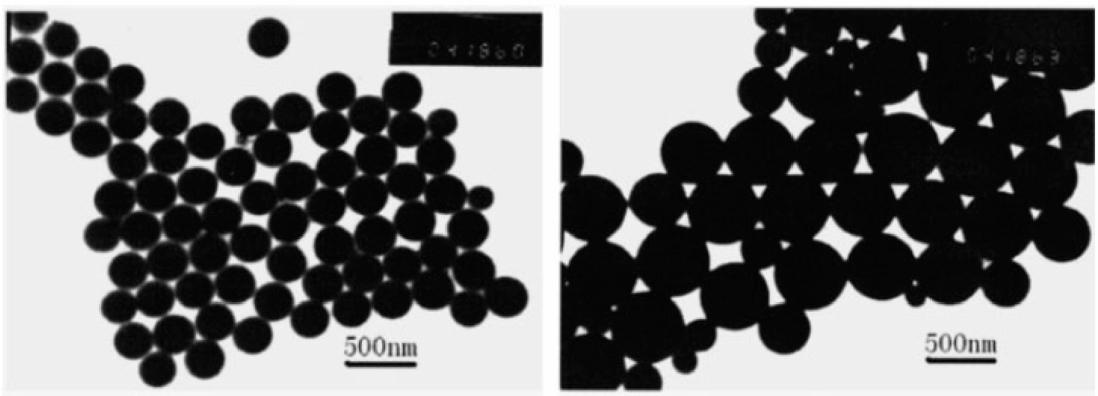

Fig. 6. Images of polystyrene particles obtained in emulsion polymerization with (left) and without (right) microwave irradiation (Xu, et al., 2005).

Next to the already existing applications described in 2.2, other potential applications discussed in the literature include:

- rapid depolymerization of polyamide-6 (carpets recycling, Klun and Krzan, 2000)
- rapid curing of polyurethane (Julien and Valot, 1985) and epoxy resin Degamber and Fernando, 2004).

3. What are the development and application issues?

3.1 Technology development issues

(In Table 4 list and characterize the essential development issues, both technical and non-technical, of the technology under consideration. Also, provide your opinion on how and by whom these issues should be addressed)

Table 4. Technology development issues

Issue	Description	How and by whom should be addressed?
Engineering & design concepts for commercial-scale devices	MW reactors have been developed on the laboratory scale, so far. New design concepts are needed of commercial-scale continuous flow reactors, which would also address the issues of energy efficiency, low penetration depth of the microwave (integrated mixing devices?) and the way of irradiation. This may need the development of <u>new geometries of magnetrons</u> as well. Also, <u>materials issues</u> are of importance here (reactor walls and elements transparent to the MW, etc.)	R&D projects carried at the universities, in collaboration with equipment manufacturers and producers of industrial-scale magnetrons
Modeling and scale-up methodologies	Research in the field of MW reactors was almost entirely dominated by chemists and attracted only very little attention of chemical engineers, so far. Robust and reliable mathematical models of the microwave reactors have to be developed, together with the scale-up methodologies for those devices.	R&D projects carried at the universities, in collaboration with equipment manufacturers and producers of industrial-scale magnetrons
Control systems for commercial-scale MW reactors	Proper control of the chemical reactions carried out in the MW reactors is of crucial importance for the reliability and safety of the operation. In most cases the MW generator has to be used in and intermittent way, to avoid overheating and possible temperature runaways.	R&D projects carried at the universities, in collaboration with MW equipment vendors

3.2 Challenges in developing processes based on the technology

(In Table 5 list and characterize the essential challenges, both technical and non-technical, in developing commercial processes based on the technology under consideration. Also, provide your opinion on how and by whom these challenges should be addressed)

Table 5. Challenges in developing processes based on the technology

Challenge	Description	How and by whom should the challenge be addressed?
Small irradiation depth of the microwave versus the required scale of production	The irradiation depth of the microwave in water medium does not exceed 2 cm. This brings an important question concerning the maximum production capacity achievable in a microwave reactor. To illustrate the issue: in a single-tube flow reactor of 3 cm I.D. operating at 0.5 m/s liquid flow velocity, the maximum production capacity could reach ca. 10,000 t/year. Possible (partial) solutions to this problem could be for	This challenge should be addressed in the R&D projects on engineering & design concepts for commercial-scale MW reactors (pos. 1 in Table 4).

	example: integrated intensive mixing devices in larger diameter tubes (to renew the irradiated liquid layer continuously) or multitube reactor designs.	
Narrow applicability of microwaves in terms of the type of media used in the process	MW are only applicable to the substances with specific dielectric properties, containing dipoles and/or ions. In other systems introduction of an extra MW-sensitive solvent or a MW-absorbing solid phase can be considered	Feasibility studies at the end-user for each specific case
Limitations related to the constructional materials of MW reactors	Tubes in MW flow reactors have to be made of materials transparent to the microwave radiation, which excludes metals as constructional materials. This may put constrains on the conditions, in which such reactors can be applied on industrial scale.	This challenge should be addressed in the R&D projects on engineering & design concepts for commercial-scale MW reactors (pos. 1 in Table 4).
Safety	Operations involving microwaves require certain safety precautions as MW irradiation is dangerous for the human health. Current technology of MW ovens enables a fully safe use of these devices.	Process development has to involve the vendors of the MW-devices.

4. Where can information be found?

4.1 Key publications

(Provide the list of key publications in Table 6)

Table 6. Key publications on the technology

Publication	Publication type (research paper/review/book/ report)	Remarks
H. Julien and H. Valot, Polyurethane curing by a pulsed microwave field, 1985, Polymer, 506-510	Research paper	
Y. Imai, Recent advances in synthesis of high-temperature aromatic polymers, 1996, Reactive and Functional Polymers, 3-15.	Review	
R. Correa, et al., Emulsion polymerization in a microwave reactor, 1998, Polymer, 39, 1471-1474	Research paper	Styrene polymerization
U. Klun and A. Krzan, Rapid microwave induced depolymerization of polyamide-6, 2000, Polymer, 41, 4361-4365	Research paper	
D. Bogdal, et al., Microwave assisted synthesis, crosslinking and processing of polymeric materials, 2003, Adv. Polym. Sci., 163, 193-263	Review	
B. Degamber and G.F. Fernando, A comparative study on the cure behaviour of thermal- and microwave-processed epoxy resin, 2004, J. Near Infrared Spectrosc., 12, 221-231	Research paper	
J. Pielichowski, et al., Microwave-assisted synthesis of unsaturated polyester resins, 2004, Polimery, 49, 763-766	Research paper	
S. Velmathi, et al., A rapid eco-friendly synthesis of poly(butylene succinate) by a direct	Research paper	

polymerization under microwave irradiation, 2005, Macromol. Rapid Commun., 26, 1163-1167		
Z-S. Xu et al., Monodisperse Polystyrene microspheres prepared by dispersion polymerization with microwave irradiation, 2005, J. Polymer Sci. Part A, 43, 2368-2376	Research paper	
C. Koopmans, et al., Microwave-assisted polymer chemistry: Heck-reaction, transesterification, Bayer-Villinger oxidation, oxazoline polymerization, acrylamides and porous materials, 2006, Tetrahedron, 62, 4709-4714	Review	
F. Wiesbrock, et al., Microwave-assisted polymer synthesis: stat-of-the-art and future perspectives, 2004, Macromol. Rapid Commun., 25, 1739-1764	Review	
C. Ludlow-Palafox and H. Chase, Microwave pyrolysis of plastic wastes, 2006, in: “Feedstock recycling and pyrolysis of waste plastics” (J. Scheirs, Ed.), J. Wiley, 569-594	Book chapter	
D. Bogdal and A. Prociak, Microwave synthesis of polymeric materials: scale-up and commercial aspects, 2007, Chemistry Today, 25 (2), 42-44	Review	
C. Zhang, et al., Recent developments in microwave-assisted polymerization with focus on ring-opening polymerization, 2007, Green Chem., 9, 303-314	Review	
R. Hoogenboom and U. Schubert, Microwave-assisted polymer synthesis: recent developments in a rapidly expanding field of research, 2007, Macromol. Rapid Commun., 28, 368-386	Review	

4.2 Relevant patents and patent holders

(Provide the list of relevant patents in Table 7)

Table 7. Relevant patents

Patent	Patent holder	Remarks
WO 97/13137: Microwave assisted chemical process	CEM Corporation	
US 6455317: Method of controlling a chemical process by microwave radiation	CEM Corporation	
US 6649889: Microwave-assisted chemical synthesis instrument with fixed tuning	CEM Corporation	
US 6713739: Microwave-assisted chemical synthesis instrument with fixed tuning	CEM Corporation	
WO 03/089133: Microwave-assisted synthesis instrument with controlled pressure release	CEM Corporation	
US 6403939: Microwave apparatus and methods for performing chemical reactions	Personal Chemistry I Uppsala AB	
US 6614010: Microwave heating apparatus	Personal Chemistry I Uppsala AB	
EP 1521501: Microwave heating	Personal Chemistry I	

device	Uppsala AB	
WO 2004/054707: Method and apparatus for control of chemical reactions	Personal Chemistry I Uppsala AB	
WO 2004/054705: Microwave heating system	Personal Chemistry I Uppsala AB	
CN 1140251, CN 1080867C: Multifunctional industrial microwave system	XU Y	
KR20060108910: Preparation method of polymethyl methacrylate	JANG HM	
US 5543605: Method for coating fibres using microwave energy	AVCO Corporation	

4.3 Institutes/companies working on the technology

(Provide the list of most important research centers and companies in Table 8)

No institutes or companies are known to work specifically on the development of microwave polymerization reactors for production purposes. In Table 8 some research groups carrying out research on MW-assisted non-catalytic or homogeneously catalyzed reactions are listed.

Table 8. Institutes and companies working on the technology

Institute/Company	**Country**	**Remarks**
Friedrich Schiller University Jena (Prof. U.S. Schubert)	Germany	Broad range of research activities
Cracow University of Technology (Prof. D. Bogdal)	Poland	Focus primarily on polymer synthesis with MW
Delft University of Technology (Prof. A. Stankiewicz)	The Netherlands	Focus on integration with separation, polycondensation reactions

5. Stakeholders

5.1 Suppliers and developers

(Provide the list of key suppliers/developers in Table 9)

No suppliers of commercial-scale MW polymerization reactors are known. Manufacturers of laboratory MW reactors and manufacturers of large-scale microwave equipment for other operations (drying, heating, sintering, etc.) can be seen as potential stakeholders here. Some of these manufacturers are listed in Table 9.

Table 9. Supplier and developers

Institute/Company	**Country**	**Remarks**
CEM Corporation	U.S.A.	World's biggest developer and manufacturer of microwave

		reactors for laboratory applications (both monomode and multimode, batch and continuous)
Milestone	Italy	Manufacturer of microwave reactors for laboratory applications (both monomode and multimode, batch and continuous)
Biotage (formerly Personal Chemistry)	Sweden	Manufacturer of microwave reactors for laboratory applications (multimode, batch), mostly for synthetic, analytical and medicinal chemistry.
Anton Paar	Austria	Offers one type of multimode batch MW reactor for organic synthesis
Plazmatronika	Poland	Offers one type of multimode batch MW reactor for organic synthesis; also manufacturer of commercial-scale MW dryers
MEAC nv	Belgium	Manufacturer of commercial MW equipment for polymer processing

5.2 End users

(Describe the existing and potential end-users, other than those already listed in Table 2)

Potential group of end users includes companies operating in the polymer manufacturing and processing sector, as well as some manufacturers of the plastic consumer products.

6. Expert's brief final judgment on the technology

(maximum 5 sentences)

Microwave equipment for polymerization reactions is in embryonic stage of development and an interdisciplinary R&D effort is needed in order to arrive at significant number of commercial applications. With such an effort industrial-scale MW polymerization reactors could be developed and implemented within next 5-10 years. On the other hand, microwaves appear to be used quite frequently in the processing of polymers, rubber and plastic products, where substantial energy savings are reported.

A potentially very important aspect of the application of microwaves in the polymerization reactions is a possibility of obtaining new products with properties not achievable under conventional heating. This may also bring secondary energy savings elsewhere in the process (e.g. uniform particle size may save energy in the particle fractionation stage).