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A Review of Microwave Pyrolysis of Biomass and Waste for the Production of Energy and Fuels

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A Review of Microwave Pyrolysis of Biomass and Waste for the Production of Energy and Fuels

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POLYTECHNIQUE MONTRÉAL

PROF. FRANCO BERRUTI

Winner of 2013 PTF Group 3b Lectureship in Fluidization Award

CONGRATULATIONS on this well-deserved honour!

OUTLINE

1. Microwave pyrolysis fundamentals;
2. Temperature measurement/prediction
3. Reaction kinetics of MW-P vs. C-P
4. Energy consumption of MW-H
5. Biomass/waste to value added chemicals and energy
6. Conclusion and perspectives

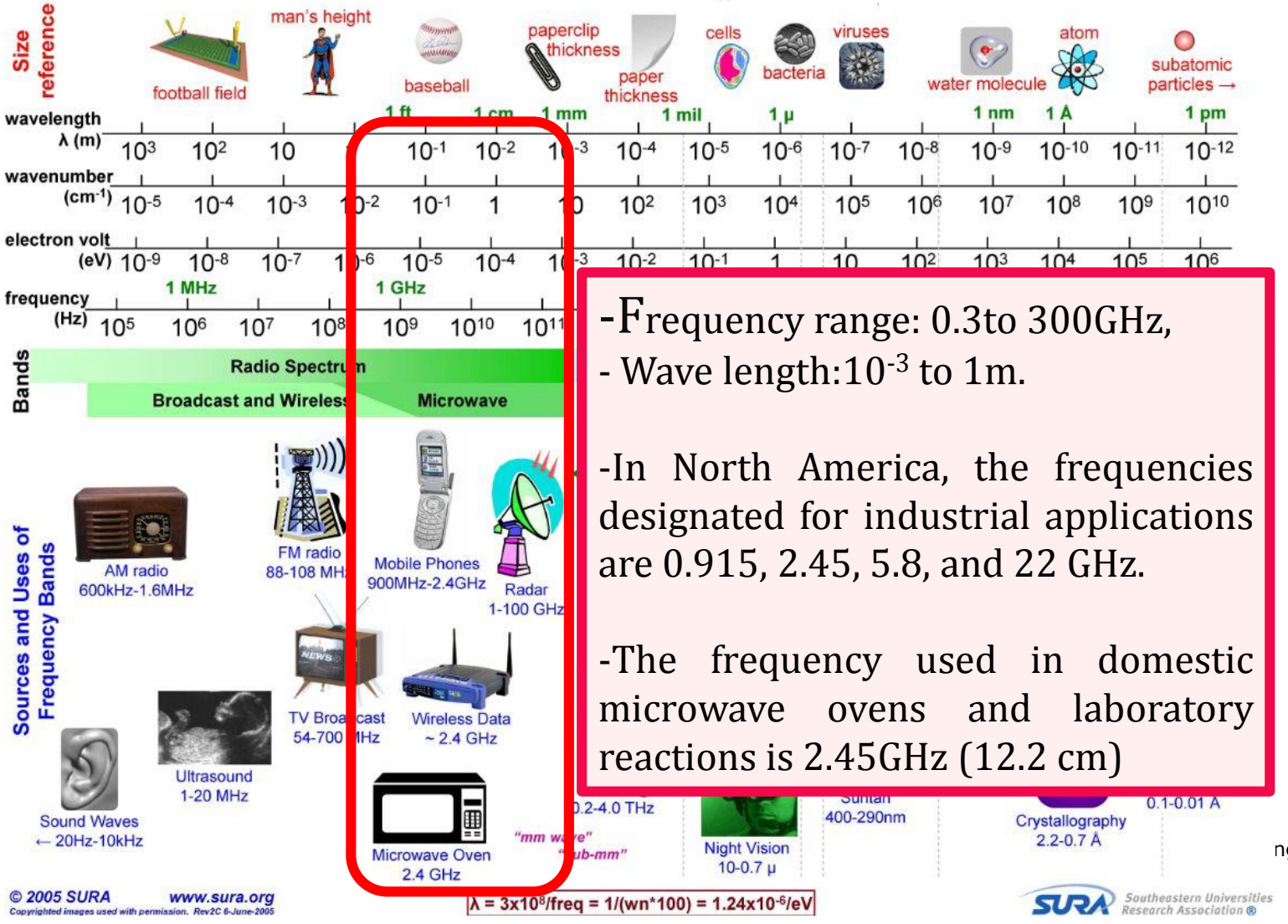
1- MICROWAVE PYROLYSIS FUNDAMENTALS

In pyrolysis technology, the needed heat energy can be provided by:

- **Heat transfer** form a heating source, it called conventional pyrolysis (C-P),
- **Heat generated** within the heated material by an electromagnetic irradiation, it called microwave pyrolysis (MW-P).

2- MICROWAVE PYROLYSIS FUNDAMENTALS

What is Microwaves?

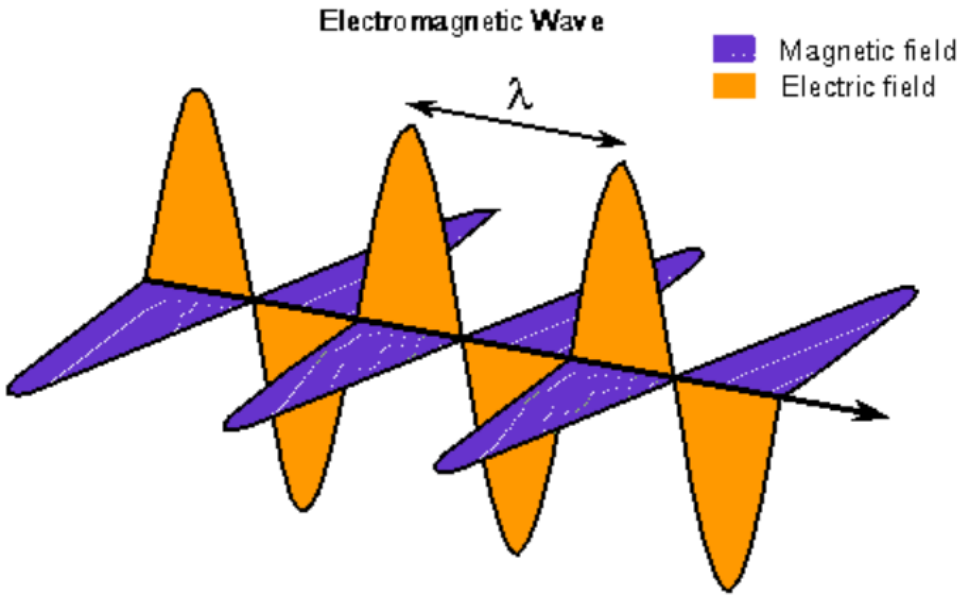


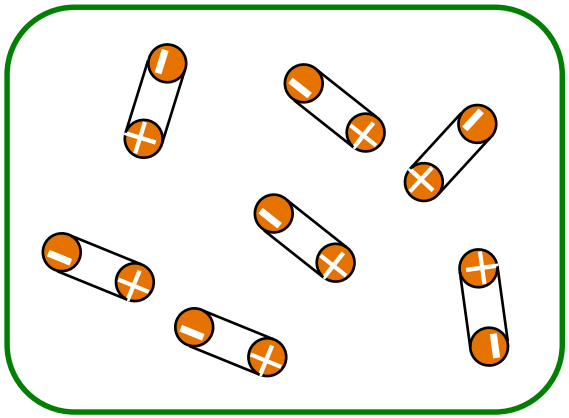
-Frequency range: 0.3to 300GHz,
- Wave length:10^{-3} to 1m.

-In North America, the frequencies designated for industrial applications are 0.915, 2.45, 5.8, and 22 GHz.

-The frequency used in domestic microwave ovens and laboratory reactions is 2.45GHz (12.2 cm)

2- MICROWAVE PYROLYSIS FUNDAMENTALS





In presence of an oscillating electromagnetic field, molecular-dipoles reorient themselves in order to be in phase with the alternating field.

THE 1ST MICROWAVE OVEN

- M. Percy Spencer working with RAYTEHON in 1945 for Microwave Radar Transmitters;
- Accidental Discovery: Melting Choclate bar;
- 1st Wicrowave Oven in 1947 (1.8 m high and 340 Kg): 3kW 60,000$

1- MICROWAVE PYROLYSIS FUNDAMENTALS

The main parameters describe the level of heat-generation inside a heated material are:

- **Dielectric constant (ε'):**

  represents the amount of electric energy that can be stored within the heated material.

- **Loss Factor (ε''):**

  represents the ability of the heated material to dissipate microwave energy.

- **Penetration depth (D_P)**:

  the depth where the magnitude of the electric field drops by a factor 1/e with respect to the surface value.

- **loss tangent ($tan\delta$)**:

  the ratio between ε'' and ε'.

1- MICROWAVE PYROLYSIS FUNDAMENTALS

Dielectric constant, dielectric loss, and loss tangent of different materials.

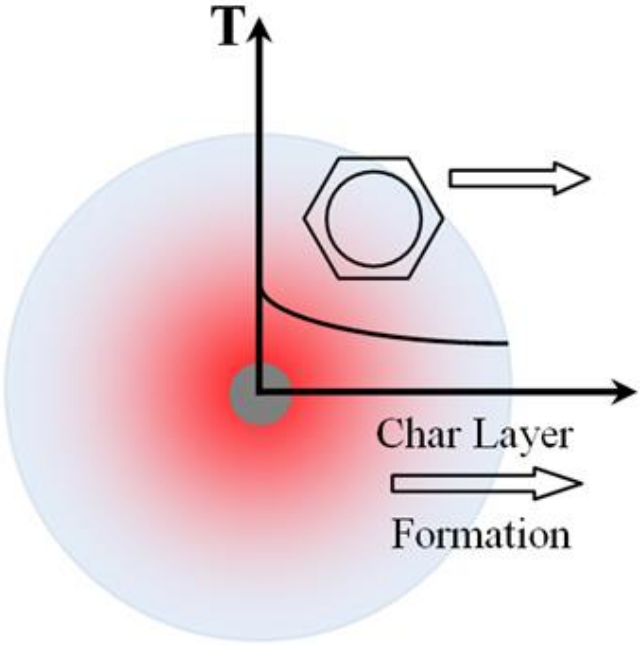
Material	ε'	ε''	$tan\delta$	Temperature	Frequency GHz
Vacuum	1.00	0	0	25C	ND
Air	1.0006	0	0	25C	ND
Glass	4.82	0.026	0.0054	25C	3
Alumina	8.9	0.009	0.00010	800C	(3.89-3.61)
Fused quartz	3.8	0.0001	0.00003	25C	2.45
Pyrex	4	0.005	0.0013	25C	2.45
Water	80.4	9.89	0.123	25C	2.45
Methanol	32.6	21.48	0.659	25C	2.45
Ethanol	24.3	22.86	0.94	25C	2.45
Silicon carbide	105	110	1.048	200C	2.45

-D. C. F. D.E. Clark, "What is microwave processing," presented at the Microwave Solutions for Ceramic Engineers, Westerville, OH 2005.
-E. W. W. L. Manoj Gupta, Wai Leong Wong, Microwaves and metals: John Wiley & sons (Asia) Pte Ltd 2007.

1- MICROWAVE PYROLYSIS FUNDAMENTALS

In addition to the previous, using of MW-H under controlled conditions would promise to eliminate several issues/limitations contrasting to conventional heating (C-H).

Wood block heated by 2.45GHz microwaves for 360s @ 2.7kW.



Therefore, MW-H would promise to improve product selectivity/quality.

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2- TEMPERATURE MEASUREMENT/PREDICTION

One of the MW-H challenges is that measuring of a transient temperature within the heated material.

Thermocouple thermometer is danger with low accuracy, for the interaction between microwaves and thermocouple metallic probe;

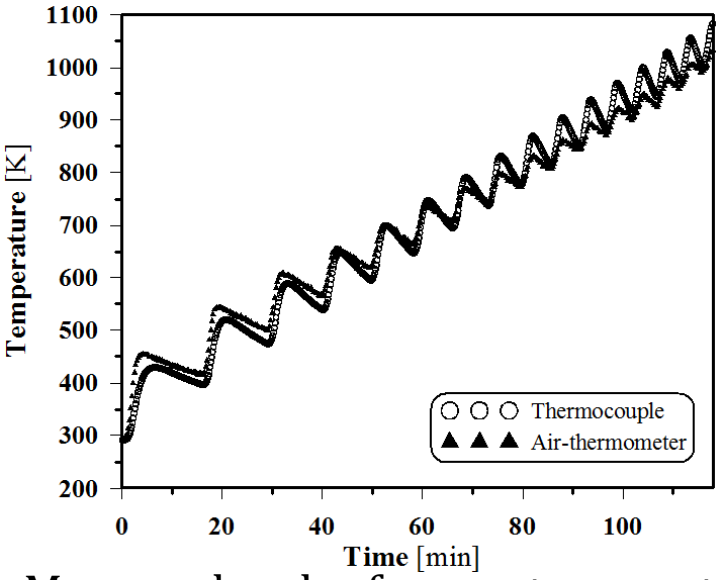
Infrared thermometer is suitable only for a very thin sample because it measures a surface temperature;

Fibre optics thermometer measures a point temperature with a limited range; in addition, it needs highly care; to avoid probe damage.

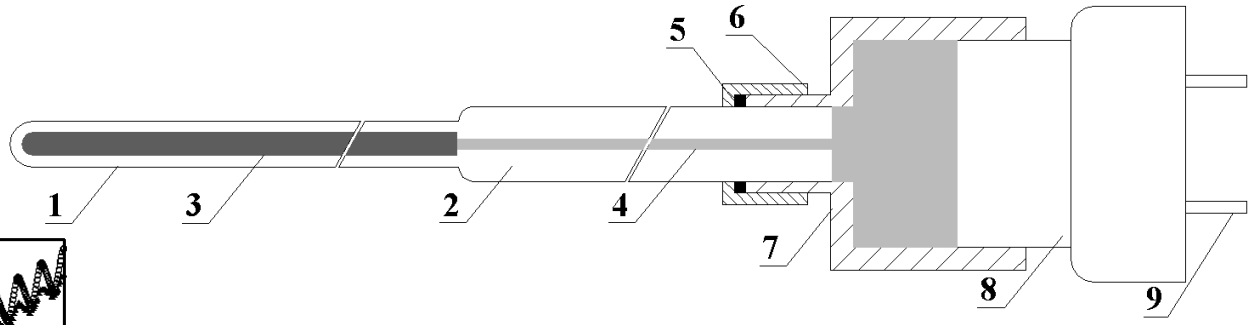
We have passed this challenge by innovating a thermometer that can be used inside a microwave oven, during the heating.

2- TEMPERATURE MEASUREMENT/PREDICTION

A novel thermometer, which can measure transient mean temperature within the heated material, has been made.



Measured and reference temperature vs. heating time



1- First tube
2- Second tube
3- Volume *Va*
4- Volume *Vb*
5- O-ring
6- Tube fitting
7- Pressure transducer fitting
8- Pressure transducer
9- Electric terminals

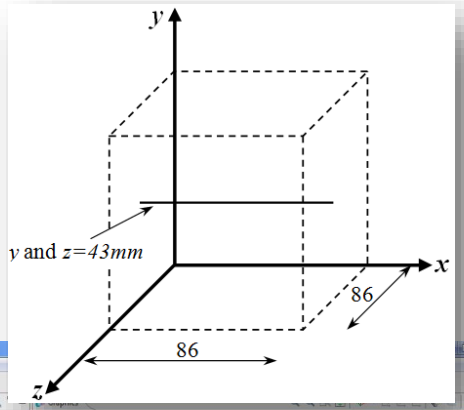
Drawing of the air-thermometer used

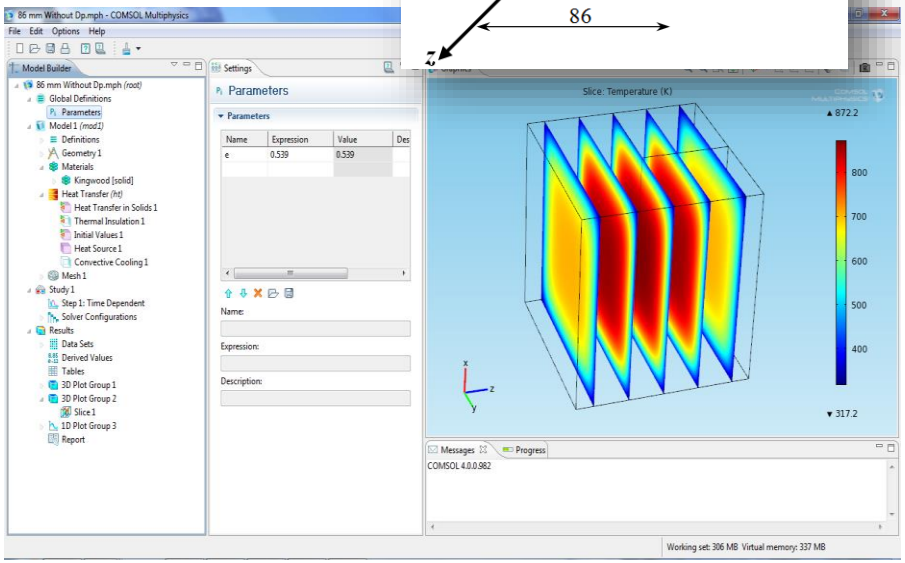
2- TEMPERATURE MEASUREMENT/PREDICTION

In addition, a 3D Mathematical model was done; to predict temperature profiles within a material exposed to MW-H.

$$k\,\nabla^2 T + \frac{P_o}{3}\sum_i \left(e^{\frac{-i}{D_p}} + e^{\frac{i-l}{D_p}}\right) = \rho C \frac{\partial T}{\partial t}$$

$(P_o)_{material} = 2.25x10^6(\varepsilon/\tan\delta)_{material}$ [W/m^3] @ 2.3kW/2.45GHz.





Assumptions:

1- Uniform distribution of electromagnetic waves.

2- All the heated materials are non-magnetic materials

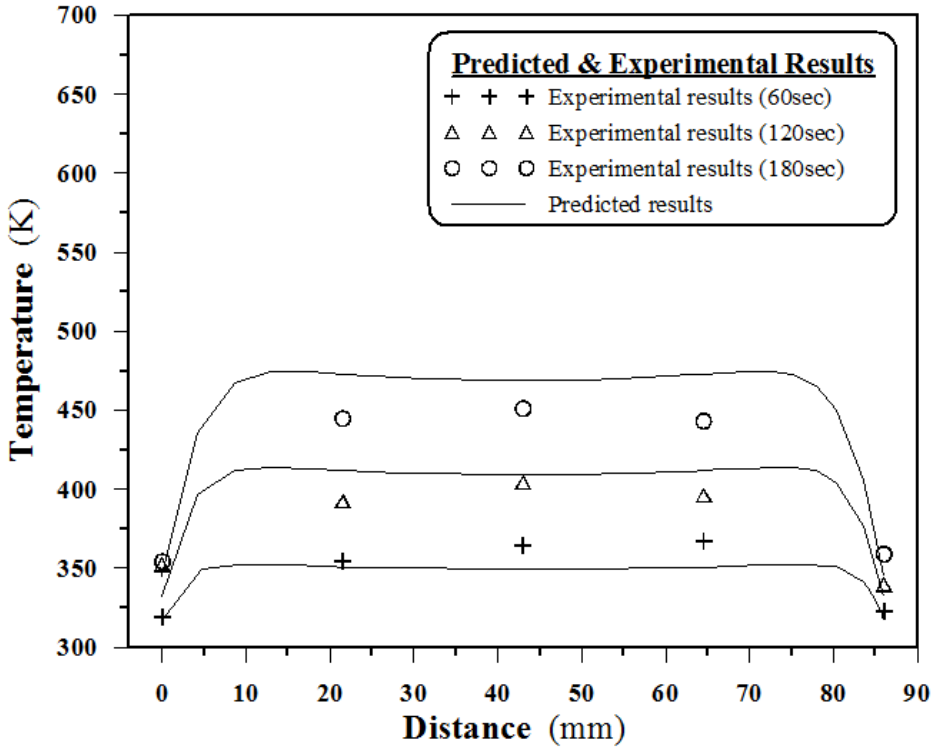
3- Variation of volume and physical/electrical properties are considered negligible.

4- Neglect any effect related to a chemical reaction.

Farag, S. and J. Chaouki., *Temperature profile prediction within selected materials heated by microwaves at 2.45GHz.* Applied Thermal Engineering, 2012. 36(0): p. 360-369.

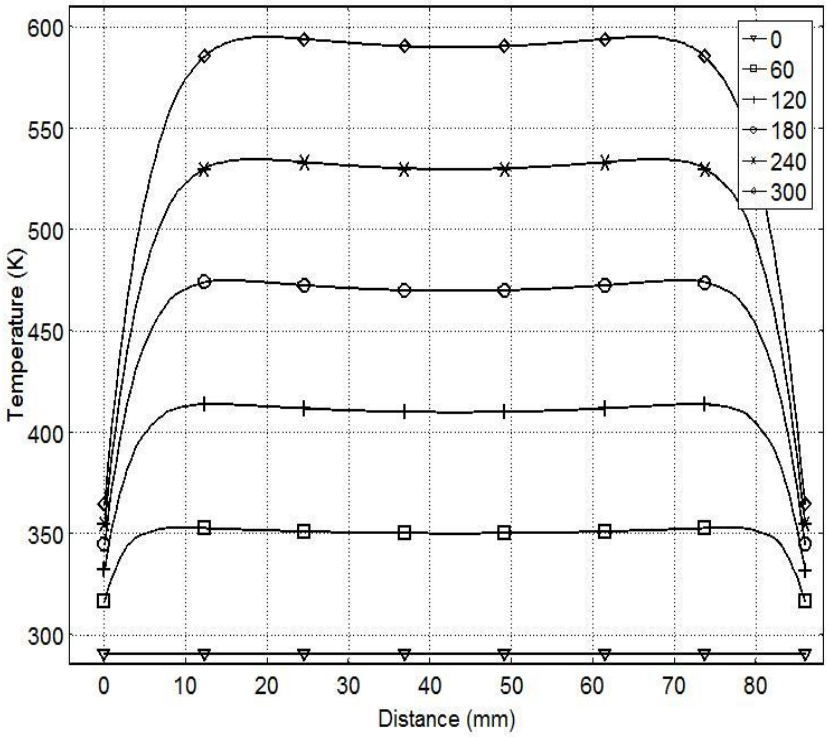
2- TEMPERATURE MEASUREMENT/PREDICTION

The average percentage relative error between the predicted and the experimental values was ±4% of the measured value from T_{amb} to 500K.

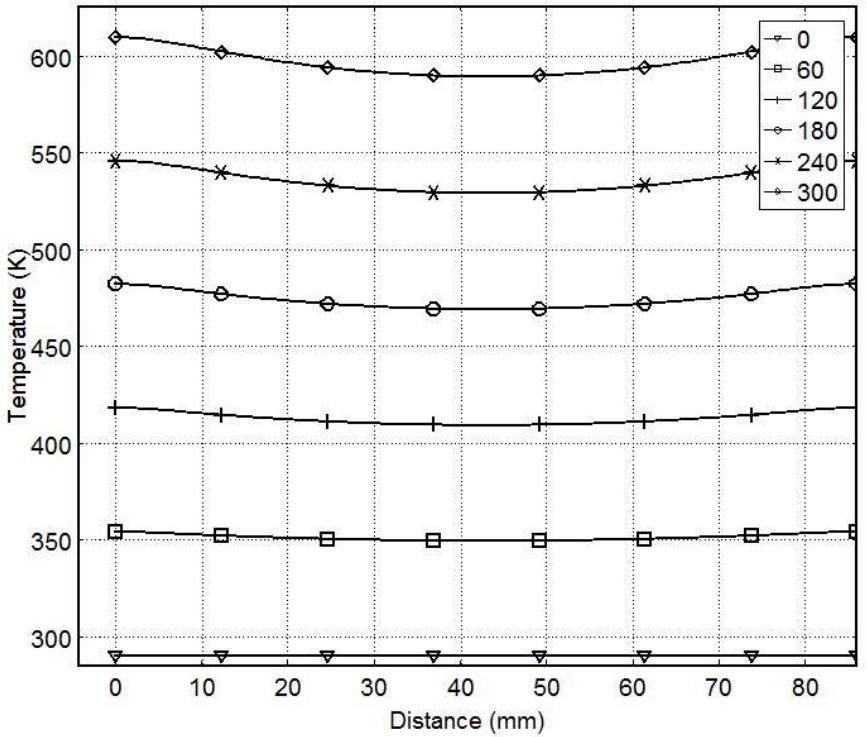


Experimental and predicted results for validation of the model for the case of Free Convection (FC)

2- TEMPERATURE MEASUREMENT/PREDICTION



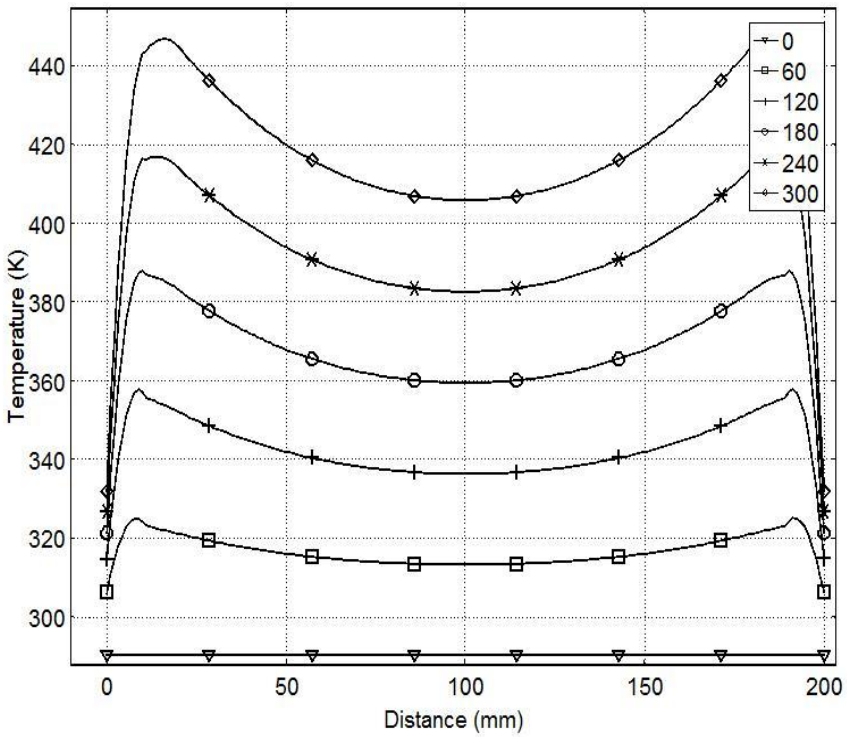
(A)



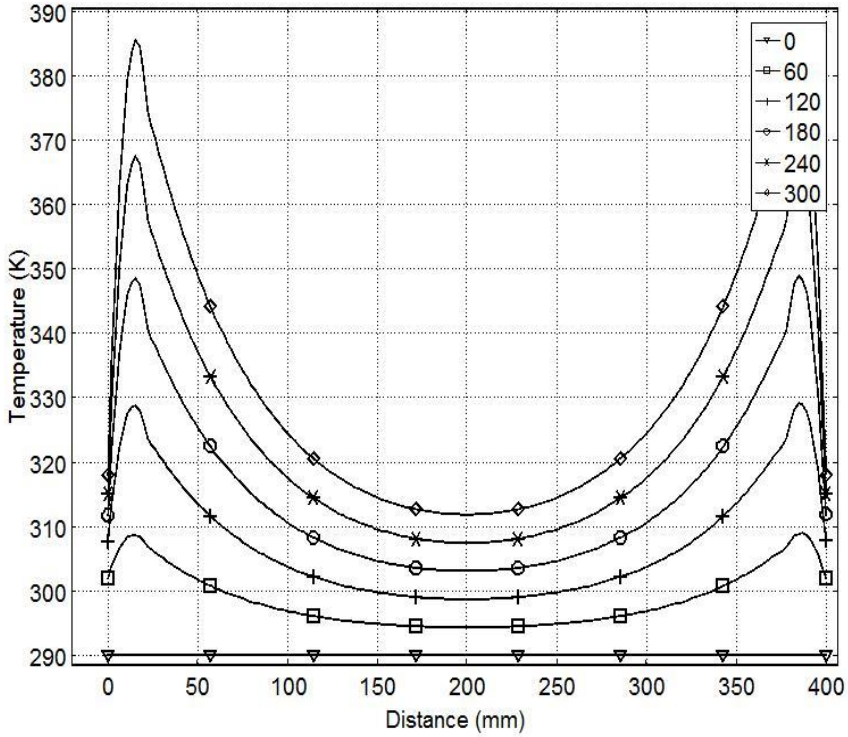
(B)

The temperature profiles on the selected line over 300s of MWH:
(A) With FC and (B) With PI. (time in seconds)

2- TEMPERATURE MEASUREMENT/PREDICTION



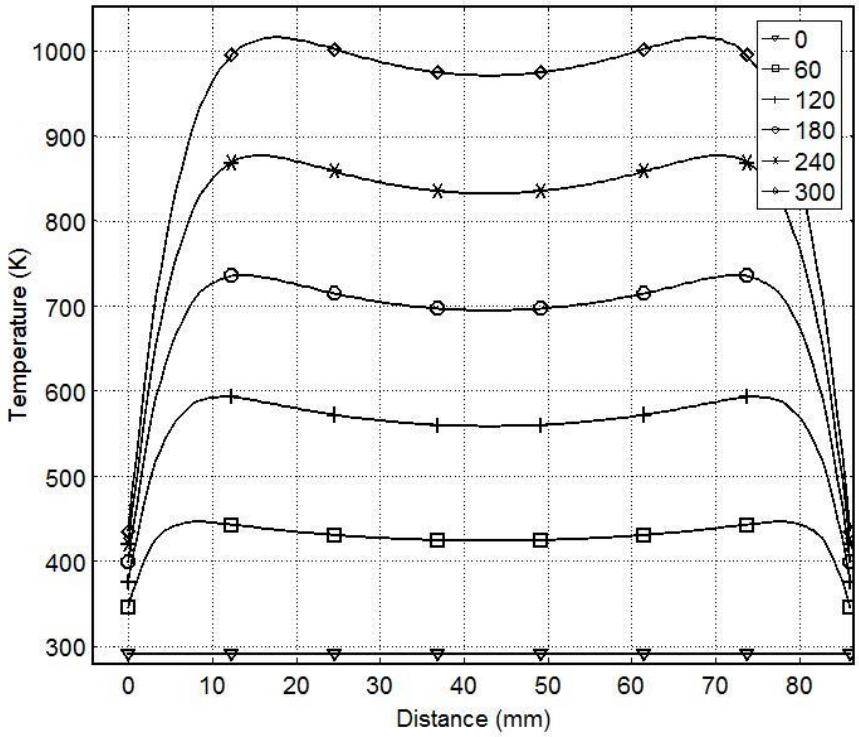
(A)

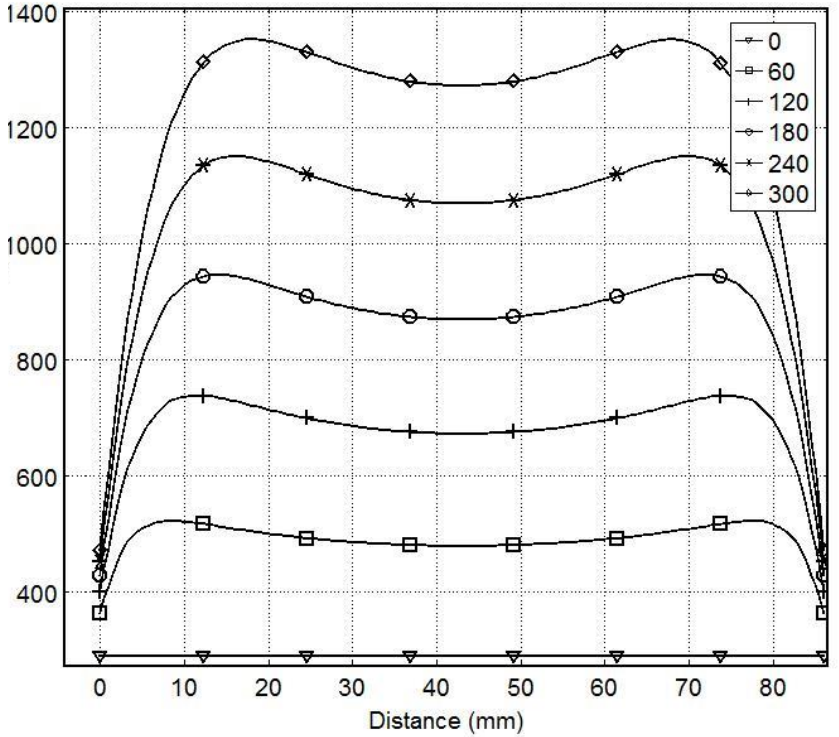


(B)

The effect of the heated material dimensions on the temperature profiles (A) 200mm and (B) 400mm of cube side length

2- TEMPERATURE MEASUREMENT/PREDICTION

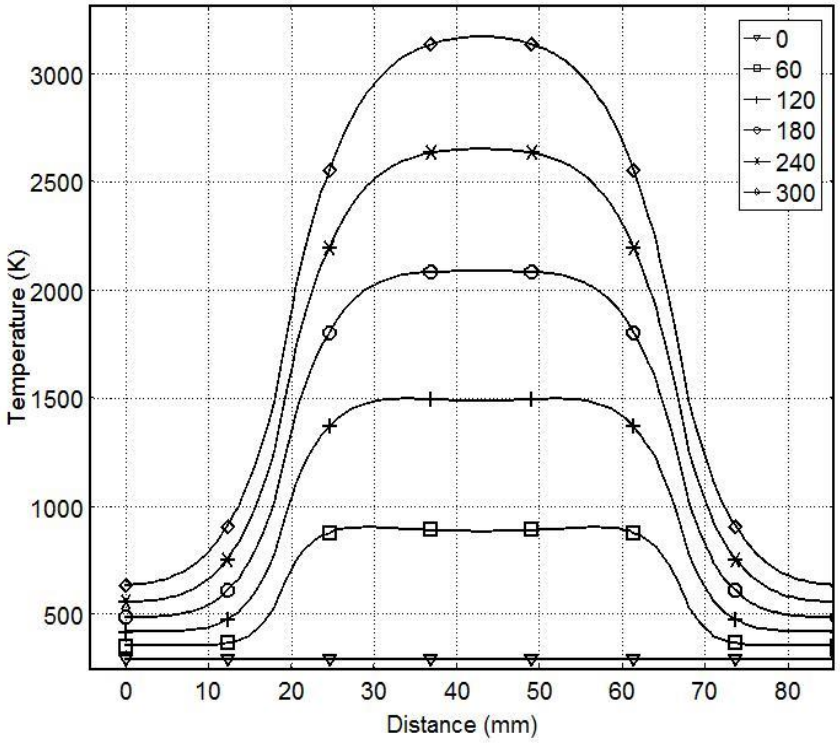




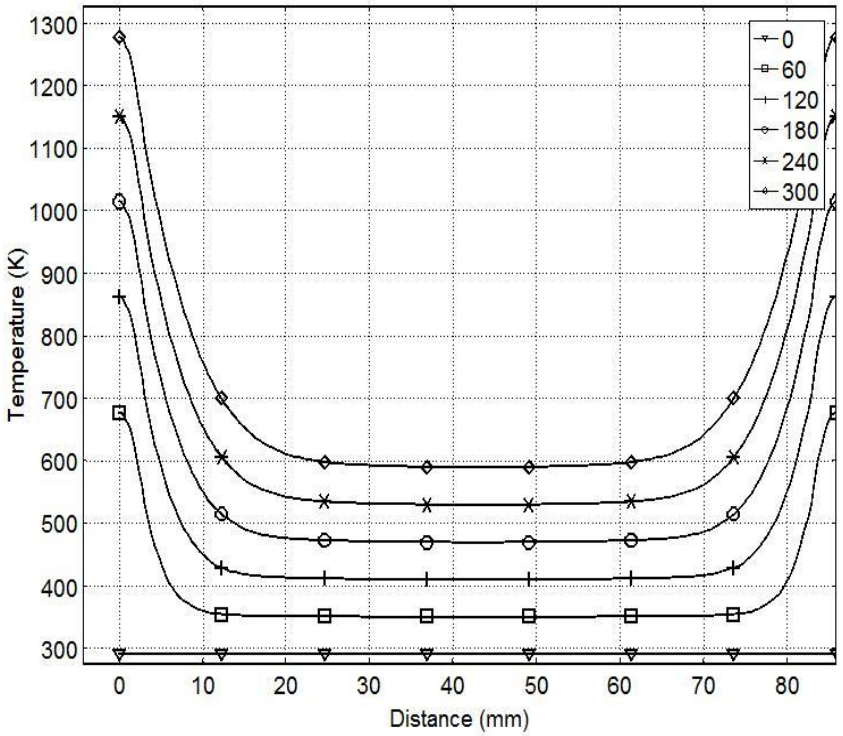
(A) (B)

The effect of substituting wood with carbon (A) 50 wt-% carbon and (B) 75 wt-% carbon

2- TEMPERATURE MEASUREMENT/PREDICTION



(A)



(B)

The effect of 125cm^3 carbon cube: (A) Embedded in the core
(B) Covered the outer surface

OUTLINE

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3- REACTION KINETICS OF MW-P VS. C-P

Selected examples regarding MW-H effects on a chemical reaction.

Author	Objective	MW-H effect
Eymir Tekin and Okur. 2011.	Dissolution of colemanite in boric acid solutions in C-H and MW-H.	Superior conversions can be obtained via MW-H.
Temur Ergan and Bayramoğlu. 2011.	Investigate the specific effects on decomposition of aqueous Potassium Persulfate.	Rate constant was about 1.1 to 1.8 times higher than that of C-H at the same temperature, depending on the MW-H power.
Chandra Shekara, et al. 2011.	Heterogeneous esterification of phenylacetic acid with p-cresol over H-β zeolite catalyst under MW-H.	MW-H was found to be superior to C-H in terms of yields, and in short reaction periods.
A. Domínguez, Menéndez ,et al. 2005.	Investigations into the characteristics of oils produced from MW-P of sewage sludge.	The oils from MW-P are more aliphatic and oxygenated than those produced by C-H at the same temperature, and they still preserve some of the functional groups from the sludge.

3- REACTION KINETICS OF MW-P VS. C-P

Selected examples regarding MW-H effects on a chemical reaction cont.

Author	Objective	MW-H effect
Zhang and Zhao. 2010.	Production of 5-hydroxymethylfurfural and furfural from lignocellulosic biomass (Corn stalk, rice straw, and pine wood) in an ionic liquid.	Increases product yield and decreases reaction time.
Budarin, Clark et al. 2009.	Preparation of high-grade bio-oils from wheat straw as a pellet using MW-P.	The produced bio-oil is rich in aromatics compared to the other produced by the conventional methods.
Guiotoku, Rambo et al. 2009	Studying of hydrothermal carbonization method of pine sawdust and cellulose.	Increases the carbonization yield.
Krzan and Zagar 2009.	Liquefaction of biomass (Poplar sawdust, wood chips, barks) with glycols using p-toluenesulfonic acid as a catalyst.	Decreases liquefaction time with minimum use of catalyst.

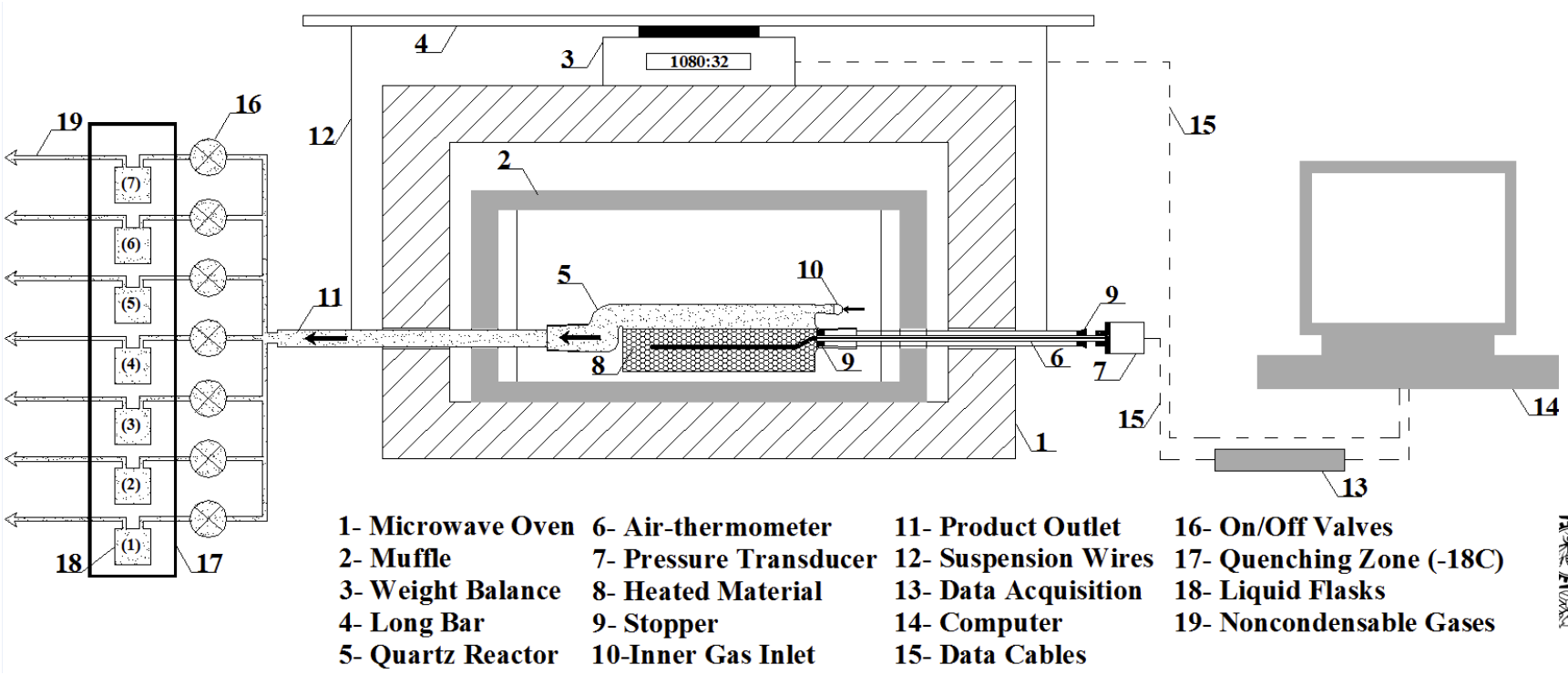
LITERATURE OBSERVATIONS

Selected examples regarding MW-H effects on a chemical reaction cont.

Author	Material	Observation
Chandra Shekara, et al. 2012.	Solventless acylation of p-cresol with different carboxylic acids over BEA zeolite.	Achieves more conversion compared to CH: 50-80% in MW-H, compare to less than 20% on C-H.
Patil, Gude et al. 2011.	Transesterification of Camelina sativa oil using metal oxide catalysts under C-H as well as MW-H	The reaction rate constants are two orders of magnitude higher than those obtained with C-H.

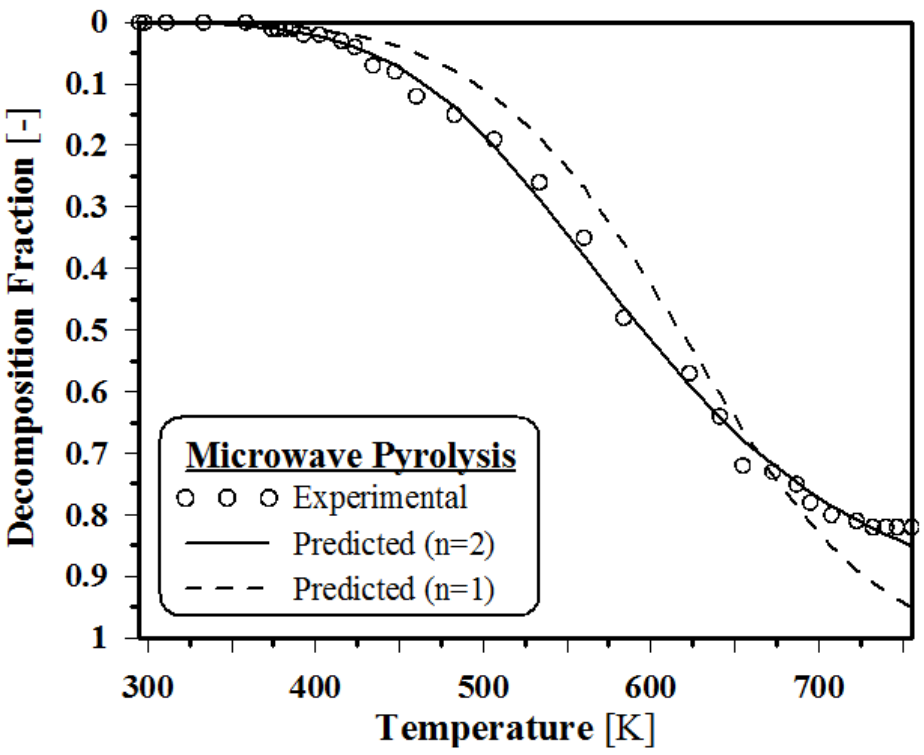
3- REACTION KINETICS OF MW-P VS. C-P

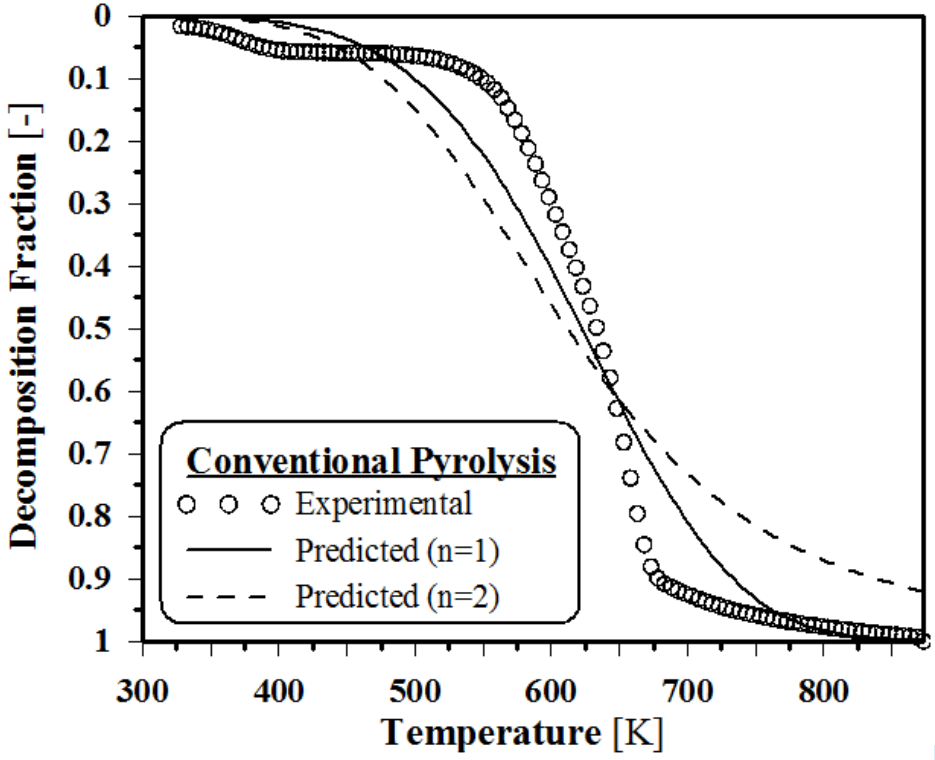
An original **MW-TGA** was built and equipped with a product manifold; to be used in kinetic purposes.



MW-TGA equipped with a product manifold

3- REACTION KINETICS OF MW-P VS. C-P





Decomposition fraction vs. temperature: experimental and predicted; (A) MW-P and (B) C-P.

Sawdust was chosen because it does not have a high resistance for thermal degradation.

3- REACTION KINETICS OF MW-P VS. C-P

The estimated kinetic parameters in MW-P and C-P

E_a kJ/mol]	k_o [min-1]	n [ ]	Deviation (%)	The process
31	683	2	2	MW-P
31	349	1	6	MW-P
31	101	1	7	C-P
31	166	2	12	C-P

MW-P may have a reaction rate faster than that of C-P, for molecular chaotic-motion resulted by the oscillating-electromagnetic-waves. Accordingly, $(k_o)_{MWP} > (k_o)_{CP}$.

3- REACTION KINETICS OF MW-P VS. C-P

Compared to the exegeses that have reported in the literature;

- Binner, Hassine et al. (1995) have reported that

> *"Analysis of the results indicates that the microwave reaction rates were 3.3-3.4 times faster.* ***It would appear that the phenomenon can be explained by a 3.3-fold increase in the Arrhenius pre-exponential factor, A, with no change in the activation energy****. The pre-exponential factor is dependent on the vibration frequency of the atoms at the reaction interface and hence it could be postulated that this might be being affected by the microwave field. Attempts to explain the increased reaction rates in terms of faulty temperature measurement were unsuccessful."*

- Temur Ergan and Bayramoğlu 2011 have documented that

> *"The experiments showed that MW energy input influences the decomposition rate of K2S2O8 in aqueous solution.* ***The reaction order is not affected, but the rate constant is influenced by MW irradiation****. The increase in the rate constant is not as high as expected when compared to previously reported values. Thus, these results neither prove nor reject the existence of a strong "specific MW" effect on chemical reactions."*

Binner, J. G. P., N. A. Hassine, et al. (1995). "The possible role of the pre-exponential factor in explaining the increased reaction rates observed during the microwave synthesis of titanium carbide." Journal of Materials Science 30(21): 5389-5393

Temur Ergan, B. a. and M. Bayramoğlu (2011). "Kinetic Approach for Investigating the "Microwave Effect": Decomposition of Aqueous Potassium Persulfate." Industrial & Engineering Chemistry Research 50(11): 6629-6637.

3- REACTION KINETICS OF MW-P VS. C-P

➢Sun, J., W. Wang, et al. (2012) have claimed that

*"Compared with a simulated conventional TGA incorporating a similar heating rate, **the activation energy in microwave-induced pyrolysis is much smaller**. This can be attributed to the internal-type heating style and a catalyst effect caused by the presence of microwave heating or microwave-metal discharges. ."*

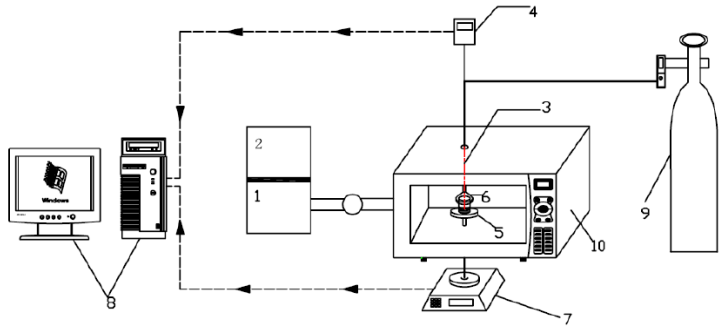
Authors of this work have done the temperature measurements by two ways:

1-Online measurement by placing a thermocouple 5 mm directly above the sample.

2- Inserting a thermocouple into the center of the sample immediately after turning off microwaves.

The used measurement technique was the main reason behind this wrong explanation.

Figure 1. Experimental set-up for microwave pyrolysis of WPCB: (**1**) microwave generation system; (**2**) microwave control system; (**3**) thermocouple; (**4**) temperature monitoring system; (**5**) PTFE tray system; (**6**) quartz container; (**7**) weighing system; (**8**) signal acquisition and processing system; (**9**) nitrogen and (**10**) microwave oven.



Sun, J., W. Wang, et al. (2012). "Kinetic Study of the Pyrolysis of Waste Printed Circuit Boards Subject to Conventional and Microwave Heating." Energies 5(9): 3295-3306.

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4- ENERGY CONSUMPTION OF MW-H

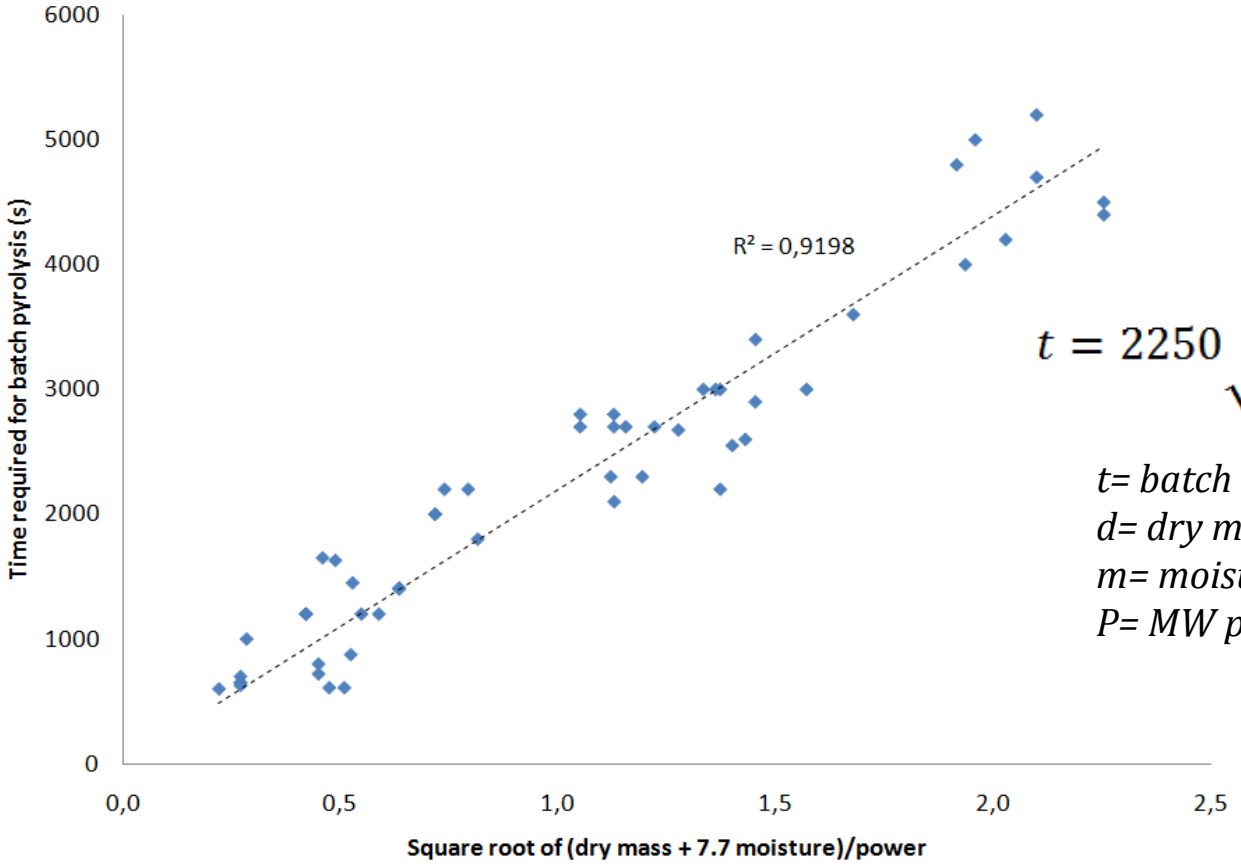
The key factors, which affect on energy consumption of MW-P are:

- **Level of microwave-to-heat conversion,** which depends on dielectric properties of the heated material. It can be improved by adding a strong microwave-to-heat converter (carbon, silicon carbide, etc.);
- **Payload dimensions,** which is limited by penetration depth of the heated material (depends on dielectric properties).

Nevertheless, exposure time and setting power could not be considered as key factors; they depend on the previous parameters.

4- ENERGY CONSUMPTION OF MW-H

Effect of payload dimensions/mass



$$t = 2250\sqrt{\frac{d + 7.7m}{P}}$$

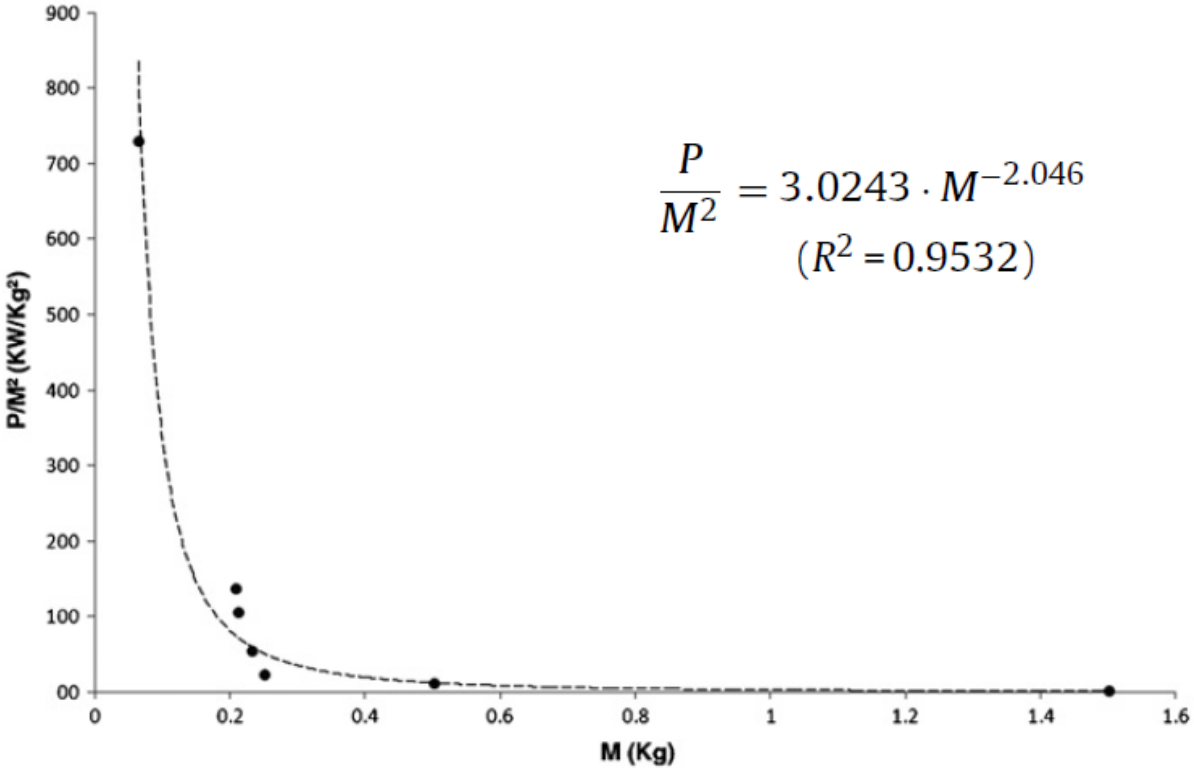
t= batch time (s)
d= dry mass (g)
m= moisture (g)
P= MW power (W)

Correlation between total batch pyrolysis time and the square root of the modified mass-power ratio.

Amr Sobhy, et al. (2013). "Simplified Global Model for Semi-Batch Microwave Pyrolysis of Lignocellulosic Biomass", will be submitted soon

4- ENERGY CONSUMPTION OF MW-H

Effect of payload dimensions/mass



$$\frac{P}{M^2} = 3.0243 \cdot M^{-2.046}$$
$$(R^2 = 0.9532)$$

Ratio between microwave power (P) and payload mass square (m^2). MW-P of waste tires.

Undri, A., S. Meini, et al. (2012). "Microwave pyrolysis of polymeric materials: Waste tires treatment and characterization of the value-added products." Journal of Analytical and Applied Pyrolysis.

4- ENERGY CONSUMPTION OF MW-H

Comparison of energy saving for conventional and microwave processes of ceramics.

	Energy saving ($X10^6$ kW.hr/yr)				
	Conventional drying	**Microwave drying**	**Conventional firing**	**Microwave firing**	**Total energy saving**
Brick and tile	56.1	28.05	198.9	19.9	207.06
Electrical porcelain	3.52	1.76	12.48	1.25	12.99
Glazes	16.63	8.3	58.97	5.89	61.37
Pottery	1.96	0.98	6.95	0.69	7.23
Refractory	10.87	5.4	38.53	3.85	40.08

W. W. L. Manoj Gupta, Eugene, "Microwave Heating," in Microwaves and metals, ed singapore: john willey, 2007.

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5- BIOMASS/WASTE TO VALUE ADDED CHEMICALS

Selected results showing different products produced by MW-H and C-P

Product and yield	Feedstock	MW-P	C-P
Gas yield (wt%)	sewage sludge[a]	33–45	22–35
	coffee hulls[b]	60–70	55–65
	glycerol[c]	55–85	47–82
Production of syngas (H2 +CO) [L g^{-1} feedstock]	sewage sludge[a]	0.50–0.56	0.25–0.40
	coffee hulls[b]	0.41–0.62	0.17–0.36
	Glycerol[c]	0.34–0.93	0.20–0.87
	wheat straw	54% total gas volume (37% H_2)	<40% total gas volume
	corn straw	54% total gas volume (35% H_2)	<40% total gas volume
Production of CO2 [L g^{-1} feedstock]	sewage sludge[e]	0.02–0.15	0.04–0.18
	coffee hulls[b]	0.20–0.34	0.45–0.65
	glycerol[c]	0.00–0.04	0.00–0.15
Oil yields [wt%]	waste oil[d]	85	46–80
	sewage sludge A[e]	10.3	3.1
	sewage sludge B[f]	2.2–4.0	0.9
Petroleum fractions in wasteoil pyrolysis (%)	waste automotive oil[g]	Gasoline (C_4–C_{12}): 69	Gasoline (C4–C12): 40
		Kerosene (C_{11}–C_{15}): 16	Kerosene (C11–C15): 18
		Diesel (C_{15}–C_{19}): 15	Diesel (C15–C19): 13
		Heavy oil (>C_{19}): 4	Heavy oil (>C19): 34

[a] T=1000C, moisture content: 0–81 wt%, two different feedstocks. [b] T=500–1000 C. [c] T ¼ 400–900C. [d] T ¼ 550C. [e] T=1000 C. [f] T ¼ 1000C, two different microwave devices and microwave absorbers. [g] MW-P vs. C-P using an electric oven.

Luque, R., J. A. Menendez, et al. (2012). "Microwave-assisted pyrolysis of biomass feedstocks: the way forward?" Energy & Environmental Science 5(2): 5481-5488.

5- BIOMASS/WASTE TO VALUE ADDED CHEMICALS

Selected results showing different products produced by MW-H and C-P

Authors	Feedstock	Yield		MW-P	C-P
Dominguez, et al. 2008.	**sewage sludge A81, A0, AN71, and AN0** **Temp 1000C** A: Aerobic sludge; AN: Anaerobic sludge; 81: moisture content=81%; 71: moisture content=71%; 0: Dried sludge.	**Char**		**59.9 - 57.8 – 57 - 55.3**	**69.3 – 74.1 – 67.7 – 68.7**
		Liquid		**3.7 – 1.8 – 4 – 2.1**	**2.4 – 1.6 – 2.1 – 2.3**
		Gas		**36.4 – 40.4 – 39 – 42.6**	**28.3 – 24.3 – 30.2 - 29**
		Gas Product	**H2 +CO**	**94.1 – 92.5 – 87.9 – 87.9 [vol%]**	**76.2 – 73.3 – 68.8 – 69.6 [vol%]**
			HC	**2.2 – 3.4 – 5.3 – 4.9 [vol%]**	**12.7 – 15.2 – 15.9 – 17.7 [vol%]**
			HHV	**12.8 – 13.1 – 13.6 – 13.4 [MJ/m3]**	**15.2 – 16.3 – 16.3 – 17.4 [MJ/m3]**
Dominguez, et al. 2007.	**coffee hulls** **Temp: 500, 800, and 1000C**	**Char**		**30.2 – 25.5 – 22.7**	**29.2 – 25.3 – 24.2**
		Liquid		**7.9 – 9.19 – 8.58**	**13.6 – 11.8 – 11.3**
		Gas		**61.9 – 65.3 – 68.7**	**57.2 – 62.9 – 64.6**
		Gas Product	**H2**	**35.94 – 38.15 – 40.06 [vol%]**	**9.28 – 25.84 – 29.85 -**
			CO	**25.8 – 29.28 – 32.75 [vol%]**	**20.62 – 20.87 – 23.05**
			CO2	**28.42 – 22.7 – 17.73 [vol%]**	**56.58 – 39.14 – 32.08**
			HHV	**12.5 – 14.0 – 15.5 [MJ/m3]**	**6.6 – 10.5 – 12.7 [MJ/m3]**

Domínguez, A., Y. Fernández, et al. (2008). "Bio-syngas production with low concentrations of CO2 and CH4 from microwave-induced pyrolysis of wet and dried sewage sludge." Chemosphere 70(3): 397-403.

Domínguez, A., J. A. Menéndez, et al. (2007). "Conventional and microwave induced pyrolysis of coffee hulls for the production of a hydrogen rich fuel gas." Journal of Analytical and Applied Pyrolysis 79(1–2): 128-135.

5- BIOMASS/WASTE TO VALUE ADDED CHEMICALS

Selected results showing different chemical compounds extracted by MW-H

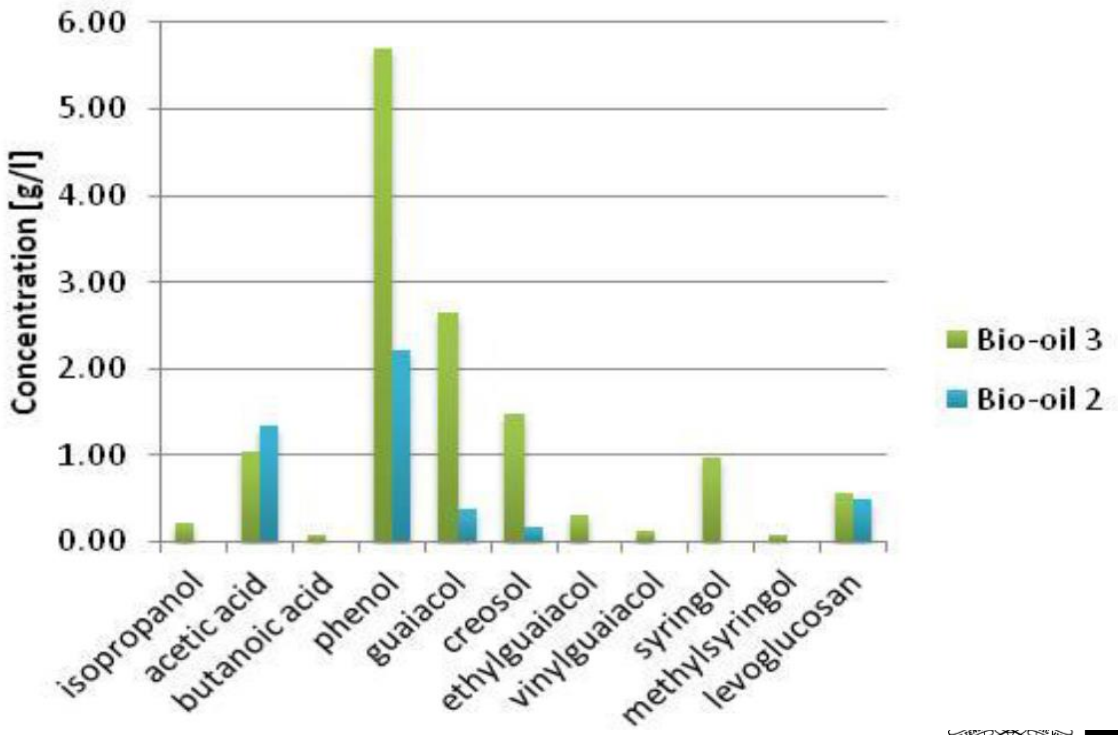
Authors	Material	Product Yield-%		Extracted Chemicals		
				Compound	Area-%	Wt.% of liquid
Quan Bu, et al. 2012.	**Douglas fir +activated carbon (AC)[a]**	Liquid	26.97	Phenol	37.72	10.17
		Gas	42.97	Phenolics	55.31	14.92
		Solid	30.06	Guaiacols	2.42	0.65
Ren, Lei et al. 2013	**Sawdust pellet**	Liquid [b]	54	Phenolics	9	4.9
		Gas[b]	19	Guaiacols	51	27.5
		Solid[b]	27	Sugars	3	1.62
				Furans	19	10.26
				Hydrocarbons	0	0
		Liquid [c]	39	Phenolics	24	9.36
		Gas[c]	29	Guaiacols	37	14.43
		Solid[c]	32	Sugars	8	3.12
				Furans	12	4.68
				Hydrocarbons	4	1.58
Undri, A., S. Meini, et al. (2012).	**waste tires[d]**	**Liquid**	**39.3**	Aromatics	29.1 % vol.	12.7 [ml/g]
		Gas	9	Paraffins	53.7 % vol.	23.45 [ml/g]
		Solid	50.7	Olefins	17.2% vol.	7.51 [ml/g]
				Oil density	0.89 [g/ml]	
				Oil Viscosity	2.36 [cp]	
				Oil HHV	43 [MJ/kg]	
				Solid HHV	34 [MJ/kg]	

[a]623K, 4min, AC to biomass 4:1, 120g, and 700W. [b]15min, 100g, and 700W, and Without torrefaction.
[c]15min, 100g, and 700W, and With torrefaction. [d]3kW, 1501g, 70min; asssume liquid density=1g/cm^3

5- BIOMASS/WASTE TO VALUE ADDED CHEMICALS

Conventional pyrolysis of lignin (in progress).

Product	C-P	MW-P
Bio-oil1 (from 25 to 300^{oC})	6%	
Bio-oil2 (from 300 to 00^{oC})	9%	
Bio-oil3 (from 400 to 50^{oC})	19%	
Total Bio-oil	**34%**	**37%**
Bio-char	**23%**	**38%**
Gas	**43%**	**25%**
Guaiacol [g/l]	**3**	**23**
Phenols [g/l]	**8**	**20**

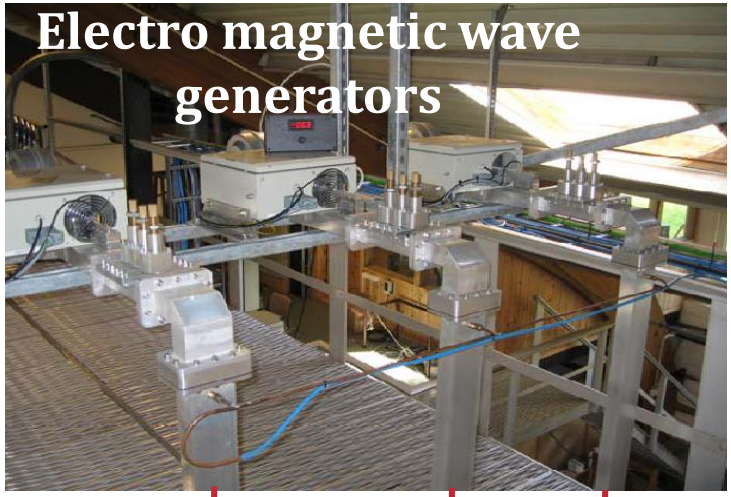


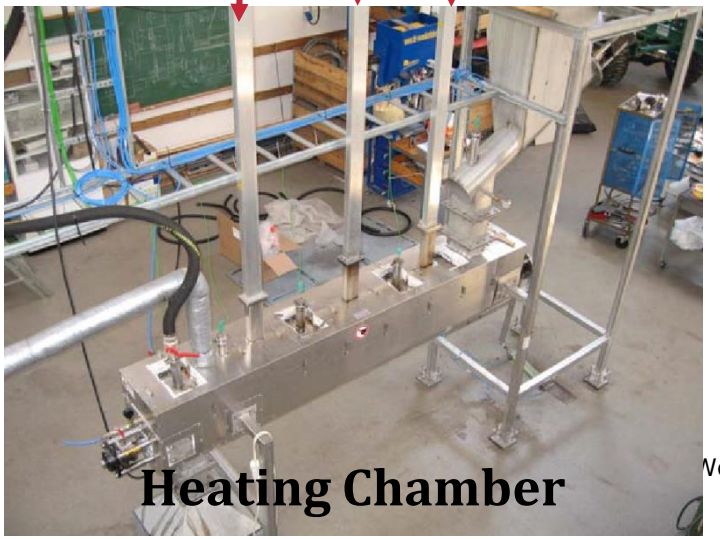
Generally, the compound extracted by MW-P have a concentration higher than that by C-H

CONCLUSION AND PERSPECTIVES

- New Chemical Reactions;
- Drying in multimode;
- MW Pyrolysis;
- Tar Gasification (Microwave Guns);
- At the nozzle feeder
- New Applications

CONCLUSION, AND PERSPECTIVES





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- With Queen's University to separate the phenolic compounds;
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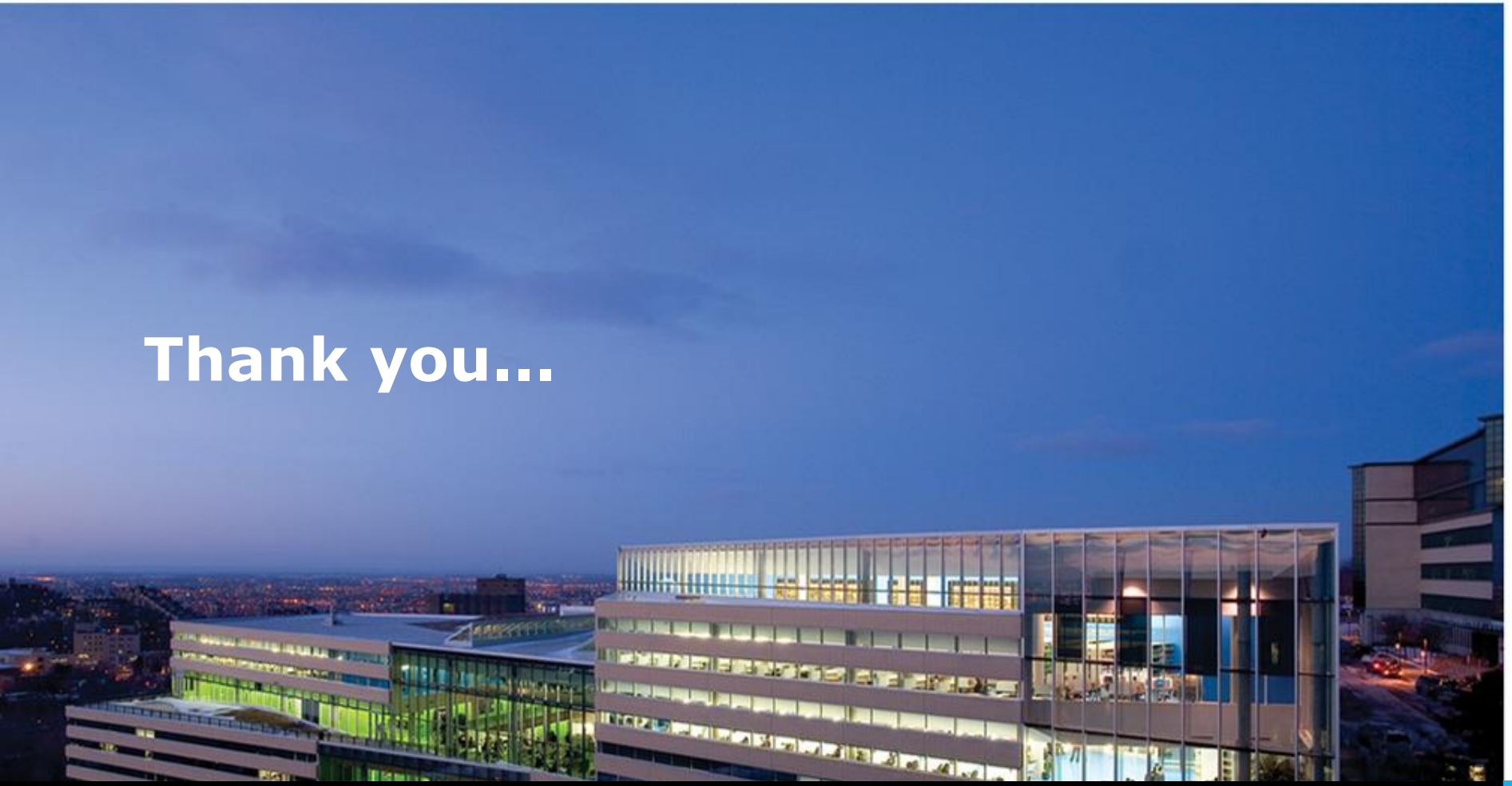

Thank you…

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