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Hydrothermal carbonization of kitchen waste

J. Malaťák, T. Dlabaja

Department of Technological Equipment of Buildings, Faculty of Engineering,
Czech University of Life Sciences Prague, Prague, Czech Republic

Abstract

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Hydrothermal carbonization is a suitable method for energy and material recovery of wet heterogeneous kitchen waste. The paper examines the ability of the process to produce stable, energy-rich material without harmful by-products from lunch leftovers, raw potatoes, creamy yogurt and raw onions. Results of the batch experiments confirm the hypothesis that waste processing results in homogenous energy-rich (> 24 MJ/kg) and carbon-rich (> 63 % wt.) material. The biochar of creamy yogurt reaches the highest lower-heating value of 31.75 MJ/kg. In terms of energy use and emission concentrations, all samples meet legal requirements for incineration in combustion devices. Phytotoxicity tests prove the harmlessness of the liquid by-product for agricultural purposes.

Keywords: biochar; hydrochar; wet pyrolysis; biomass; heating value; stoichiometry

Energy utilization of kitchen waste (KW) defined as animal by-product is still at the crossroads, not only in the Czech Republic. The aim of the waste management should be substantial reduction in land-filling through the development of energetic and material utilisation of biodegradable waste through lower environmental impact processes. There are three basic treatment technologies nowadays: waste disposal at landfills, composting and anaerobic digestion. But a few others are undergoing intensive research. The examined hydrothermal carbonization (HTC) of kitchen wastes brings good results both for solid and liquid outputs.

KW is defined by the Waste Catalogue of the Czech Republic under No. 20 01 08. The KW from restaurants, canteens and catering establishments has the potential to spread biological pathogens and infectious diseases (e.g. swine flu, foot-and-mouth disease, and diarrhoea). KW constitutes a considerable amount of biodegradable waste and it is necessary to process it further in accordance with Act No. 264/2011 Coll (Law amending the Law No. 185/2001 Coll., on waste and amending certain other laws, as amended). Everyone is obligated to ensure waste recovery prior to its disposal. Dumping of KW into sewer networks through garbage disposal units is forbidden without permission, as is the feeding of livestock with kitchen waste. KW is a subset of raw or cooked food materials. The study of the European Commission estimates annual food waste generation in the EU27 at approximately 89 Mt, or 179 kg per capita in 2006. Food waste produced by the Food Service/Catering sectors (thus kitchen waste) represents about 14% of the total or about 12.3 Mt, an average of 25 kg per capita for the EU27 (Monier et al. 2010).

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HTC of biomass is a wet moderate thermochemical process that occurs under autogenous pressures. The first experiments were carried out already in 1913 by Friedrich Bergius, German Nobel laureate. HTC was rediscovered by prof. Mark Antonietti about 10 years ago. The process is recommended for the treatment of wet biomass, which is decomposed by a series of simultaneous reactions, including hydrolysis, dehydration, decarboxylation, aromatization and recondensation (Libra et al. 2011). The transformation of the macromolecular structure of the original biomass is affected by reaction temperature (170–300°C), pressure (1–3 MPa), residence time (0–100 h), pH (acidity), initiation and polymerization reagents and the weight ratio of the solid and liquid phases in the input mixture. The result is a porous, brittle and homogeneous powder-like product, so-called biochar or hydrochar, which can have various uses (Antonietti, Titirici 2010; Titirici, Antonietti 2010; Funke, Ziegler 2011). It is a high carbon and energy dense material, which is similar to lignite coal. Few studies focused on the carbonization of food wastes. Berge et al. (2011) and Lu et al. (2012) evaluated the carbonization of rabbit food and Hwang et al. (2012) carbonized dog food. Resulting solid samples had high carbon (45–93% of initial carbon) and energy (15–30 kJ/g dry solids) contents. Ramke et al. (2009) and Lu et al. (2012) report that the hydrochar energy content correlates well with the carbon content of the solids, and the hydrochar resulting from the carbonization of food waste contained a high energy content (> 25 MJ/kg). Small carbon fractions remain in the liquid and gas-phases. Carbon dioxide is the predominant gas detected, with methane, ethane, propane, propene, butane and furan also detected (Lu et al. 2012). Lu et al. (2012) also characterize an interesting phenomenon – the carbonization fraction associated with food waste initially increases, and then abruptly decreases. The initial increase is likely a result of initial feedstock solubilisation.

Lu et al. (2012) examined energy balances of HTC of food waste collected from local restaurants. Results from these analyses indicated that carbonization of food waste containing 5% solids results in a net loss of energy due to the energy required to heat the large mass of liquid present. Although the carbonization of food waste containing 20% solids was energetically positive, it was not more favourable than the incineration of food waste. Carbonization of received food waste (32% solids, dry weight) was energetically positive and more energetically advantageous than incineration.

The research work is focused on the hydrothermal carbonization of KW (lunch leftovers) and foodstuffs – raw potato, creamy yogurt (fat content 10%) and raw onion. An essential task of the work is to determine the basic fuel properties of the final solid products of HTC. Above all, it is the determination of elemental analysis, determining stoichiometric properties in comparison with the legal requirements for biofuels in the Czech Republic.

MATERIAL AND METHODS

Experimental measurements of HTC process were mainly based on the Prof. Antonietti's published patent "Process for converting biomass to coal-like material using hydrothermal carbonisation" in 2009. The process is recommended for the treatment of wet biomass and includes two phases – initiation and polymerization. The solids content of the raw KW varies between 15–30%. wt. and pH within the 5–7 range. In the experiments, the raw biomass was diluted to 10% wt. for the sake of good mixing and heat transfer. pH adjustment was made by adding citric acid. Solutions of 30% H_2O_2 and $FeCl_3{\cdot}6H_2O$ were chosen as polymerization reagents.

For the experiment and analysis the food samples were selected from the university dining service, which was provided by Eurest – the international catering company. The biomass was shredded by a food chopper prior to use.

To produce biochar by hydrothermal carbonization, a multifunctional pressure vessel with accessories was used – a batch reactor BR-300 (Berghof, Eningen, Germany) with a useful volume of 350 ml fitted with a thermometer, a barometer, and a hole for external pressurization. The sample was stirred using a magnetic stirrer with heating brand model Heidolph Hei-Standard. For the initial stage the temperature 215°C was selected, with a set residence time of 10 min. to determine the effect of reaction temperature on the heating value. For the polymerization phase the temperature was 190°C and the residence time in the range of 2, 4 and 10 hours. The range of residence time was used to determine the effect on the calorific value of the product. The low temperature of HTC treatment is preferred in terms of lower energy requirements

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for heating. The experiments followed the concept of biphasic hydrothermal carbonization. Between phases the pressure vessel was cooled to 70°C and opened to add polymerization reagents into the biomass test sample. Solid and liquid components of the final product were separated by filtration through filter paper and then dried in an oven at 55°C for 24 h to remove residual moisture. Subsequently samples were exposed to air and left for one month to lie on a common table in a laboratory. Their degradation was compared with samples stored in a closed desiccator in the dark.

To evaluate fuel properties of the examined samples, elemental analysis was performed. This is a fundamental of every calculation for the heat work of any combustion equipment. Individual weight percentages of carbon, hydrogen, oxygen, sulphur, nitrogen and all water in the samples were determined.

Elemental analyses were performed at the University of Chemistry and Technology Prague, in the Faculty of Environmental Technology. Amounts of carbon, hydrogen and nitrogen were assessed on the analyser CHN Elementar vario EL III (Elementar Analysensysteme GmbH, Hanau, Germany) according to ČSN ISO 29541:2012 (Solid mineral fuels – Determination of total carbon, hydrogen and nitrogen content – Instrumental method). Accuracy of the method was determined by the manufacturer for the simultaneous determination 5 mg of standard 4-amino-benzen sulphanilic acid in the module CHNS < 0.1% abs. for each component.

For chlorine and sulphur examination, samples were burned in an oxygen-hydrogen flame on Wickbold Combustion Apparatus (Koehler Instrument Co., New York, USA) according to ČSN EN 15289:2011 (Solid biofuels – Determination of total content of sulfur and chlorine). Non-combustible substances of fuels, i.e. ash content and all water content, were assessed by incineration, respectively reducing the water content of the sample. A certified moisture analyser Ohaus MB 25 (Ohaus Corporation, Parsippany, USA) was used to determine the total water content according to ČSN EN 14774-1:2010 (Solid biofuels – Determination of moisture content – Oven dry method – Part 1: Total moisture – Reference method).

The higher heating value of the examined samples was determined by using the calorimeter IKA 2000 according to ČSN EN 14918:2010 (Solid biofuels – Determination of calorific value). The Lower heating value was calculated according to EN 14918:2010 using the results of the elemental analyses of the samples.

The following stoichiometric analysis of combustion processes complements the sample characteristics, and it is the basis for any thermal calculation. It is particularly important for many problems in design practice, as well as for assessing the existing combustion apparatus. The amount of oxygen (air) required for complete combustion of fuel, the quantity and composition of fuel and the specific density of the exhaust gas are determined in these calculations (Gürdíl et al. 2009). For our stoichiometric analysis we considered the complete combustion and the reference quantity of oxygen in the combustion gases. Calculation of the air consumption and the amount of flue gas were determined in this work in an analytical manner, i.e. according to elemental analysis using stoichiometric equations. All volumes and weights of combustion air and flue gases are given for normal conditions, i.e., at t = 0°C and pressure p = 101.325 kPa, and the reference oxygen content in the flue gases O_r = 11%.

Valid emission regulation of the Czech Republic No. 415/2012 Coll. (in accordance with the EU's regulation) defines qualitative indicators for solid biofuels for combustion stationary apparatus with a total rated thermal input of up to 5 MW inclusive. These demands on the quality of solid fuels are shown in Table 1 and are compared with the meas-

Table 1. Regulation No. 415/2012 Coll. requirements for quality of solid fuels from biomass

Qualitative indicator	Unit	Limit values according to the total rated input of the combustion device	
		≤ 0.3 MW	> 0.3–5 MW
Water	% wt.	< 15	< 20
Qualitative indicator in anhydrous state			
Lower heating value	MJ/kg	> 15	> 13
Ash content	% wt.	< 10	< 25
Chlorine content	mg/kg	< 10,000	< 10,000

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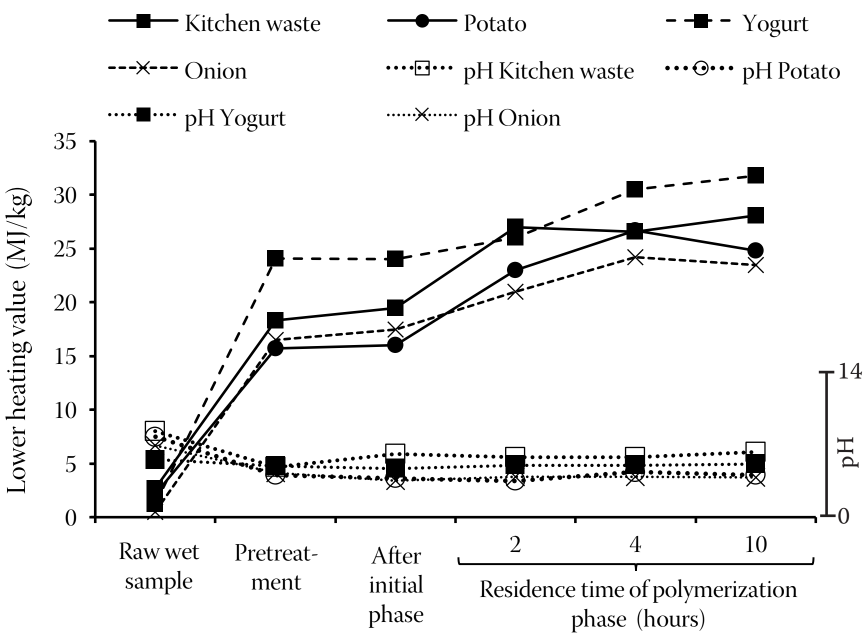

Fig. 1. Parameters of the hydrothermal carbonization of kitchen waste, potato, yogurt and onion on processing time

ured parameters of the examined samples of kitchen waste, potato, creamy yogurt and onion. For the evaluation of the measured values, basic statistical methods were used.

RESULTS AND DISCUSSION

Process parameters of HTC (Fig. 1) confirm the effect of increasing the temperature to increase the lower heating value (LHV). The reaction time has the greatest influence on the LHV in the first 4 h of the second phase. Results of the batch experiments confirm the hypothesis that HTC of wet and heterogeneous food waste results in a homogenous energy-rich (> 24 MJ/kg) and carbon-rich (> 63% wt.) material.

The initial increase, then subsequent decrease and increase in the energy yield is observed for kitchen waste. It is likely a result of simultaneous feedstock solubilisation and char formation. Different patterns are observed for raw potatoes and onions, which reach their max. value in the residence time of 4 hours.

The resulting values of elemental analysis for KW (lunch leftovers) (HTC 190°C, 4 h) and raw potato (HTC 190°C, 10 h) and for biochar are shown in Table 2. The resulting values of elemental analysis for creamy yogurt (fat content 10%) (HTC 190°C, 10 h) and raw onion (HTC 190°C, 4 h) and for biochar are shown in Table 3.

The most important results of the elemental analysis are, in terms of energy use and emission concentrations, the content levels of ash, sulphur, chlorine and nitrogen in the examined samples. All concentrations of sulphur are very low. The worst is the biochar of onion with 0.55% wt. Similarly, the majority of sulphur is transformed into a gas state as SO_2 or SO_3 during combustion. Emission of sulphur for thermal equipment using solid fuels from renewable sources does not usually infer any problem with regards to limit values, as was proved with the examined samples. The decisive factor for sulphur concentration in biofuels may be its corrosive behaviour.

The concentration of nitrogen was decreased by HTC to approximately half for KW and yogurt. Another important element is chlorine, which is transformed during combustion largely to a gaseous state. Higher concentration is also observed in the sample of kitchen waste. The importance of chlorine lies on one hand in the emissions of HCl – and the possible effect on the formation of polychlorinated dibenzo/dioxins and furans (PCDD/F), and on the other hand in the corrosive effects of these elements, or their compounds (Malaťák, Passian 2011).

Other results of elemental analysis lie within the optimal parameters for using those biofuel samples in combustion apparatus.

The most important factor for the thermal utilization of solid biofuels is the water and ash content in the fuel. The content of water in all raw samples

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Table 2. Elemental analysis raw biomass and biochar of kitchen waste and raw potato

Sample of raw biomass	Kitchen waste		Raw potato	
	dried sample	original sample	dried sample	original sample
Water (% wt.)	0.00	72.85	0.00	76.80
Ash (% wt.)	3.07	0.83	4.61	1.07
Carbon (% wt.)	46.22	12.55	41.21	9.56
Hydrogen (% wt.)	6.92	1.88	6.39	1.48
Nitrogen (% wt.)	2.39	0.65	1.40	0.32
Sulphur (% wt.)	0.00	0.00	0.00	0.00
Oxygen (% wt.)	41.40	11.24	46.39	10.76
HHV (MJ/kg)	19.46	4.94	16.78	4.35
LHV (MJ/kg)	18.36	2.75	15.69	2.15
Sample of biochar	Biochar of kitchen waste		Biochar of potato	
	dried sample	original sample	dried sample	original sample
Water (% wt.)	0.00	2.76	0.00	4.40
Volatile matter (% wt.)	–	69.17	–	48.34
Non-volatile matter (% wt.)	–	26.55	–	45.41
Ash (% wt.)	1.56	1.52	1.93	1.85
Carbon (% wt.)	68.86	66.78	68.79	65.77
Hydrogen (% wt.)	7.98	7.76	5.09	4.87
Nitrogen (% wt.)	1.31	1.28	2.53	2.42
Sulphur (% wt.)	0.19	0.19	0.12	0.12
Oxygen (% wt.)	19.39	18.86	21.41	20.47
Chlorine (% wt.)	–	0.85	–	0.10
HHV (MJ/kg)	–	28.57	–	26.00
LHV (MJ/kg)	27.57	26.58	25.97	24.82

HHV – higher heating value; LHV – lower heating value

is high, which subsequently affects the fuel heating value. For processed samples the amount of water is very low, which results in a positive contribution to the LHV. The resulting products contain less than 4.42% wt. (biochar of onion) with LHV above 24 MJ/kg.

The amount of ash significantly affects the thermal properties of the examined solid samples and subsequently affects both the selection and the adjustment of combustion equipment. The resulting biochar has a low ash content, which does not exceed concentrations above 3% wt.

Another requirement for combustion is the permissible amount of water in the solid biofuel, which is set up to 15% wt. for apparatus with a nominal input power under 0.3 MW. Unlike the previous requirement, this is met in all the examined samples.

The limiting quality indicator for energy use of the examined samples is ash content. For combustion apparatus with nominal heat input between 0.3–5 MW, up to 25% wt. is permitted in the fuel. This indicator was exceeded by all products. The concentration of chlorine meets the requirements of the Regulation No. 415:2012.

The most important parameter is the total LHV of the samples before and after treatment. For all examined samples there was a high increase of LHV. The cause of the high increase of LHV dur-

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Table 3. Elemental analysis of raw biomass and biochar of creamy yogurt and raw onion

Sample of raw biomass	Creamy yogurt (10% fat)		Raw onion	
	dried sample	original sample	dried sample	original sample
Water (% wt.)	0.00	85.65	0.00	89.96
Ash (% wt.)	5.76	0.84	4.48	0.45
Carbon (% wt.)	53.93	7.74	43.834	4.40
Hydrogen (% wt.)	8.00	1.15	5.99	0.60
Nitrogen (% wt.)	4.52	0.64	1.766	0.18
Sulphur (% wt.)	0.00	0.00	0.00	0.00
Oxygen (% wt.)	27.79	3.98	43.93	4.41
HHV (MJ/kg)	25.19	3.56	17.61	2.75
LHV (MJ/kg))	24.11	1.22	16.51	0.397
Sample of biochar	Biochar of yogurt		Biochar of onion	
	dried sample	original sample	dried sample	original sample
Water (% wt.)	0.00	2.99	0.00	4.42
Volatile matter (% wt.)	–	81.83	–	50.57
Non-volatile matter (% wt.)	–	12.28	–	42.95
Ash (% wt.)	2.99	2.90	2.15	2.06
Carbon (% wt.)	71.30	69.17	65.76	62.86
Hydrogen (% wt.)	10.00	9.73	4.91	4.70
Nitrogen (% wt.)	2.68	2.60	2.74	2.62
Sulphur (% wt.)	0.19	0.19	0.55	0.53
Oxygen (% wt.)	12.71	12.33	23.56	22.52
Chlorine (% wt.)	–	0.09	–	0.29
HHV (MJ/kg)	–	33.95	–	25.31
LHV (MJ/kg)	32.73	31.75	25.29	24.17

HHV – higher heating value; LHV – lower heating value

ing the processing is a large reduction of moisture in the fuel. The biochar of creamy yogurt reached LHV of 31.75 MJ/kg.

The resulting values of the stoichiometric analysis indicate good thermal emission parameters of examined HTC products in Table 4. As follows from the stoichiometry of the examined samples, the parameters of calorific value, water content and energy density influence the selection and design of the combustion equipment. First of all, the amount of water in the raw biomass requires different combustion conditions than for the processed samples of biochar. Such biomass absolutely cannot be recommended for combustion.

Oxygen is a problematic part of fuel because it binds hydrogen, and also partly carbon, to hydroxides, water and oxide. Problems with nitrogen (in the form of amines and proteins in the fuel) and chlorine lie in their interaction with the conversion device, especially thermal ones (Gürdíl et al. 2009). Therefore determined values of the stoichiometric analysis can be used for other necessary calculations of thermal efficiency and heat loss of combustion apparatus, but mainly serve to control and optimize the combustion apparatus.

One of the advantages of the HTC product against wet biomass is the possibility for long-term storage. For that reason biochar samples were exposed to the

Table 4. Stoichiometric analysis of raw biomass and biochar samples (O_r – oxygen content)

		Kitchen waste	Potato	Creamy yogurt	Onion
Sample of raw biomass					
Theoretical amount of air	(kg/kg)	1.61	1.15	1.11	0.52
	(m^3/kg)	1.24	0.88	0.86	0.40
Theoretical amount of dry flue gases	(kg/kg)	3.68	3.22	3.13	2.56
	(m^3/kg)	1.20	0.87	0.82	0.40
Theoretical concentration of carbon dioxide in dry flue gases	(% wt.)	12.51	10.89	9.06	6.31
	(% vol.)	19.34	20.43	17.53	20.55
Dry flue gases converted to a reference oxygen content O_r = 11%					
Carbon dioxide in the flue gases	(kg/kg)	0.46	0.35	0.28	E0.16
	(m^3/kg)	0.23	0.18	0.14	0.08
Water vapour in the flue gases	(kg/kg)	1.03	1.00	1.05	1.00
	(m^3/kg)	1.22	1.19	1.27	1.22
Nitrogen in the flue gases	(kg/kg)	2.55	1.82	1.77	0.83
	(m^3/kg)	2.03	1.45	1.41	0.66
Sample of biochar					
Theoretical amount of air	(kg/kg)	9.55	8.36	10.78	7.90
	(m^3/kg)	7.36	6.45	8.31	6.09
Theoretical amount of dry flue gases	(kg/kg)	11.67	10.75	12.70	10.30
	(m^3/kg)	6.99	6.27	7.79	5.94
Theoretical concentration of carbon dioxide in dry flue gases	(% wt.)	20.98	22.44	19.97	22.38
	(% vol.)	17.71	19.44	16.47	19.61
Dry flue gases converted to a reference oxygen content O_r = 11%					
Carbon dioxide in the flue gases	(kg/kg)	2.46	2.42	2.55	2.31
	(m^3/kg)	1.24	1.22	1.29	1.17
Sulphur dioxide in the flue gases	(kg/kg)	0.00	0.00	0.00	0.01
	(m^3/kg)	0.00	0.00	0.00	0.00
Water vapour in the flue gases	(kg/kg)	1.53	1.18	1.81	1.13
	(m^3/kg)	1.51	1.14	1.82	1.09
Nitrogen in the flue gases	(kg/kg)	15.14	13.28	17.12	12.54
	(m^3/kg)	12.07	10.59	13.64	10.00
Oxygen in the flue gases	(kg/kg)	2.44	2.13	2.75	2.02
	(m^3/kg)	1.70	1.49	1.92	1.41

air and left to lie for one month on a common table in a laboratory. Their degradation was compared with samples stored in a closed desiccator in the dark. Moisture contents shown in Table 5 were very similar and relevant samples showed no visual differences.

Utilisation of the liquid by-product for agriculture purposes was examined by the phytotoxicity test. The length of the reaction decreased the negative effect of the germination capacity of the liquid state. Results are shown in Table 6.

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Table 5. Differences in moisture content (% wt.) between biochar samples after one month stored in desiccator and samples kept outside

Biochar samples	Kitchen waste			Potato			Yogurt			Onion		
HTC polymerisation time (h)	2	4	10	2	4	10	2	4	10	2	4	10
Desiccator	1.92	2.89	2.97	5.05	4.85	4.92	3.57	2.56	0.59	3.88	3.57	3.81
Out	1.82	2.91	3.51	5.40	5.27	5.40	4.22	3.50	1.20	3.95	3.65	3.78

values are averages of three measurements for each sample; HTC – hydrothermal carbonization

Table 6. Effect of liquid phase on germination capacity by phytotoxicity test

Sample	No. of plants	No. of germinated plants	Germination capacity (%)
KW (10 h): 10% solution	23	23	100.0
KW (4 h): 10% solution	21	21	100.0
KW (2 h): 10% solution	21	20	95.2
KW (10 h)	21	13	61.9
KW (4 h)	21	5	23.8
KW (2 h)	24	4	16.7

KW – kitchen waste (hydrothermal carbonization polymerisation time)

CONCLUSION

The research deals with a topical issue of energetic and material utilisation of municipal biodegradable waste. HTC meets the legal requirements for the treatment of KW defined as animal by-products. The LHV of KW's biochar increases with longer residence time. HTC is a suitable method both for reducing the water content of material and gaining a more valuable product which is easy to store. All samples of KW's biochar meet requirements of legal regulations for combustion apparatus with a nominal input power over 0.3 MW.

The future of the HTC process lies in a continually operating system with well managed heat exchange. In the case of wet waste it is necessary to find an appropriate balance between the energy input (for heating) and the product value (biochar and its quality). Quality of biochar can be influenced by the process parameters. HTC is suitable for treatment of hazardous wet materials, or for reworking other outputs which have problems with practical application and are quickly perishable. KW's biochar reaches the lower heating value over 24 MJ/kg with the carbon content over 63% wt. A solution of 10 % liquid by-product is usable for agricultural purposes.

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Corresponding author:

Assoc. Prof. Ing. Jan Malaťák, PhD., Czech University of Life Sciences Prague, Faculty of Engineering, Department of Technological Equipment of Buildings, Kamýcká 129, 165 21 Prague 6-Suchdol, Czech Republic; e-mail: malatak@tf.czu.cz