doi: 10.17221/59/2013-RAE

Hydrothermal carbonization of stabilized sludge and meat and bone meal

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Abstract

Malaťák J., Dlabaja T. (2015): **Hydrothermal carbonization of stabilized sludge and meat and bone meal**. Res. Agr. Eng., 61: 21–28.

Hydrothermal carbonization is one of suitable methods for energy recovery of sewage sludge and meat and bone meal. The task of the article is to determine appropriate hydrothermal carbonization process conditions and their impact on the quality of the final product – so called biochar or hydrochar. Parameters of the two main phases – initiation and polymerization – were monitored. The basic fuel properties of the final solid products of hydrothermal carbonization were determined. To produce biochar by hydrothermal carbonization, multifunctional pressure vessel with accessories was used – a batch reactor BR-300. Process parameters of hydrothermal carbonization confirm the effect of increasing temperature to increase the lower heating value (LHV). Neither calorific values of meat and bone meal (17.22 MJ/kg), nor calorific values of digested stabilized sludge (12.14 MJ/kg) showed a significant increase after undergoing processing. The effect of reaction temperature on the LHV of the final product is significantly higher than that of residence time. The results show that the main factor affecting LHV of the fuel sample is the final amount of ash. Unlike the meat and bone the hydrothermal carbonization of the stabilized wastewater sludge is one of the effective processing methods for subsequent energy use.

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

Energy utilization of wastewater treatment sludge is still questionable, not only in the conditions of the Czech Republic. There are three basic treatment technologies nowadays: sludge disposal at landfills, thermal treatment and composting. But the hydrothermal carbonization is the method with biggest development at present.

For combustion of sludge without any additions there are combustion facilities available operating on the principles of open-hearth furnaces, fluidized bed and deck combustion equipment. Furthermore, rotary kilns and cyclone furnaces are used to a lesser extent for sludge combustion (Werther, Obadav 1999).

Alternative technologies for thermal treatment of sewage sludge cause wide discussion. Gasification is one of these technologies, an alternative method which may reduce the amount of solid residues that must be disposed after wastewater treatment. Gasification produces a gas that can be used to generate electricity, or used for propellent units. The research of Judex et al. (2012) proves that gasification in the fluidized bed and gas cleaning with granular bed filter achieves a successful operation.

Supported by the Internal Grant Agency of the Czech University of Life Sciences Prague, Czech Republic, Grant No. 20123003.

doi: 10.17221/59/2013-RAE

Currently gaseous product mixed with biogas from the sludge can be used in a gas engine or a combustion device for heat generation (González et al. 2012). Development of gasification technology opens up further possibilities of gasification of the sludge mixture with solid biomass in the form of pellets. In the work of Seggiani et al. (2012), there are experiments carried out with a mixture of sludge and wood mass in the form of pellets. In the gasification unit at atmospheric pressure a gas with calorific value of 4.9 MJ/Nm^3 was obtained. However, there is an issue of return on investment.

Another alternative technology is pyrolysis sludge treatment. Pyrolysis technology itself for sludge treatment has been already resolved. The biggest problem arises in defining the final properties of the resulting products of the pyrolysis of sewage sludge. Zhai et al. (2012) achieved the greatest successes by means of Fourier Transform Infrared analysis in the study of intermediates in the process of pyrolysis of sludge.

The last alternative technology is hydrothermal carbonization of sludge. In the study of Kobayashi et al. (2011), hydrothermal processing is done with sewage sludge and effects of hydrothermal conditions – such as temperature, pressure, humidity and various concentrations of the final products – were monitored primarily. Solids content of the treated sewage sludge was increased to 52%, thus allowing sludge combustion without drying sludge in incinerators.

Drying is an effective way of energy utilization of sewage sludge, where reducing energy consumption is one of the major technical problems. In the study of Zili et al. (2010), a possibility of hydrothermal sludge treatment is experimentally investigated. It was found that treatment of sewage sludge by saturated steam with the temperature of 190°C and pressure of 20 MPa can dramatically improve the dehydration performance of the slurry-like product. The water content can be reduced down to about 55% by a mechanical dehydration. And after natural drying 24 h it can be reduced down to about 20%. The final product is almost odourless and can be easily used as an alternative fuel.

The research work is focused on the hydrothermal carbonization of sewage sludge, together with other waste material such as meat and bone meal, which is no longer suitable for feeding purposes. The task of the work is to determine appropriate hydrothermal carbonization process conditions and their impact on the quality of the final product – so called biochar or hydrochar. Parameters of the two main phases – initiation and polymerization – are monitored. Effects of reaction temperatures, pressures, residence times, pH, initiation reagent, polymerization reagent and the weight ratio of the solid and liquid phases of the input and the output are studied. During the process almost the complete breakdown of the macromolecular structure of the original biomass occurs, producing a porous, brittle and powder-like product that is considerably easier to use after drying than the original biomass to produce electricity, heat or fuel. The product has a significantly higher specific energy density than the original material, but during the reaction approximately one third the original biomass energy is released. Part of the energy is released as heat in exothermic processes of carbonization, and is further chemically bound within carbonaceous compounds dissolved in the liquid phase. In general, dewatering of the solid phase of the biochar product is significantly better than for original wet biomass, e.g. sewage sludge. The advantage of biochar is also high homogeneity. All these acquired properties may be of significant conceptual advantage in energy recovery compared to incineration or gasification of untreated biomass (Antonietti, Titirici 2010; Rillig et al. 2010; Titirici, Antonietti 2010; Funke, Ziegler 2011).

An essential task of the work was to determine the basic fuel properties of the final solid products of hydrothermal carbonization. Above all, it was the determination of elemental analysis, determining stoichiometric properties in comparison with the requirements for biofuels in the Czech Republic.

MATERIAL AND METHODS

Experimental measurements of hydrothermal carbonization (HTC) process were mainly based on the published patent of Prof. Antonietti "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 biomass feedstock is recommended between 5–35% wt. because of the ease of mixing and the flow of material. Drier biomass is diluted with water. pH for the initial phase is recommended in the range of 4 to 6. In the ex-

doi: 10.17221/59/2013-RAE

periments, the adjustment is made by adding citric acid. Raw biomass is activated in a closed reactor at a temperature of 19–270°C during 5–15 minutes. As a result of the heating there is an increase in pressure inside the reactor of water vapour to values of up to 3 MPa. The temperature in the second polymerization phase is lower than in the first one, preferably 170–210°C and the residence time is 1 h or more. Polymerization reagents were 30% H_2O_2 and $FeCl_3{\cdot}6H_2O$. This phase is characterized by a slightly exothermic reaction. The reaction may be stopped at different stages of the process during incomplete breakaway of water, which creates various lowest degree of petroleum products, humus or lignite. Final properties of the product can be regulated by process conditions.

For the experiment and analysis the samples are selected from a wastewater treatment plant and a veterinary sanitation institute. The samples of stabilized sludge were obtained from the Central Waste Water Treatment Plant Prague. According to the Czech Statistical Office, about 163 thousand tons of dry matter of the sludge in all wastewater treatment plants (CZSO 2012) were produced in the Czech Republic in 2011. This sludge can be utilized for energy. Conceptual solution of disposal or utilization of this material is not clearly defined in the Czech Republic.

A sample of a meat and bone meal (MBM) was taken from animal waste treatment company ASAP s.r.o., Věž, Czech Republic. This is a product with inferior quality and therefore is not suitable for the production of feeding mixture. Depending on the material classification, MBM coming from material of the first category is taken and is intended for disposal by incineration (for energy recovery) or for landfilling. MBM in this category cannot be used as organic fertilizers, soil improvers, for production of compost or biogas or digestion residues with subsequent potential use for soil application. Therefore, the only way to utilize this product is to produce energy.

To produce biochar by hydrothermal carbonization, 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 container was heated up to a temperature of 300°C and the sample was stirred using a magnetic stirrer with heating brand model Heidolph Hei-Standard (Heidolph Instruments Labortechnik, Schwabach, Germany). For the initial stage the temperatures 190°C and 215°C were selected, with an identical residence time of 10 min to determine the effect of reaction temperature on the heating value. For the polymerization phase the temperatures were 180°C and 200°C and the residence time in the range of 2–10 hours. The ranges of temperatures and residence times are used to determine the effect of these parameters on the calorific value of the product. The lower temperature is preferred in terms of lower energy requirements for heating. First, the influence of the parameters was examined purely for the initiation phase. This was followed by experiments with biphasic hydrothermal carbonization. These experiments were carried out both without pH adjustment, and the adjustment of the pH between 4 to 6. 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 followed by drying in an oven at 55°C for 24 h to remove residual moisture.

To evaluate fuel properties of the examined samples, elemental analysis was performed. This is fundamental for every calculation of the heat work of any combustion equipment. Individual weight percentages of carbon, hydrogen, oxygen, sulphur, nitrogen and all water in the samples were determined. Non-combustible substances of fuels, i.e. ash content and all water were determined by incineration or dewatering the sample.

Elemental analyses were performed at the University of Chemistry and Technology, Prague, Faculty of Environmental Technology within the internal grants of authors. The elements carbon, hydrogen and nitrogen were determined on the analyser CHN Elementar vario EL III (Elementar Analysensysteme GmbH, Hanau, Germany) according to ČSN ISO 29541:2012. Accuracy of the method is determined by the manufacturer for the simultaneous determination of 5 mg standard 4-amino-benzen sulphanilic acid in the module CHNS < 0.1% abs. for each component.

For chlorine and sulphur determination, samples were burned in an oxygen-hydrogen flame on the Wickbold Combustion Apparatus (Koehler Instrument Company, New York, USA) according to ČSN EN 15289:2011. Non-combustible substances of fuels, i.e. ash content and all water content, were determined by incineration or dewatering the sample. A certified moisture analyser Ohaus MB 25 (Ohaus

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Table 1. Requirements for quality of solid fuels from biomass

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

Corporation, Parsippany, USA) was used to determine the total water content according to ČSN EN 14774-1:2010.

Higher heating value of examined samples was determined by measuring the calorimeter IKA 2000 (IKA®-Werke GmbH & Co. KG, Staufen, Germany) according to ČSN EN 15170:2009 and ČSN EN 14918:2010. Lower heating value was determined by calculation according to ČSN EN 14918:2010 and the results of the elemental analyses of the samples were used.

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 overseeing 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 exhaust gas are determined in these calculations (Gurdíl et al. 2009). For our stoichiometric analysis we consider 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 by analytical manner, i.e. according to elemental analysis using stoichiometric equations. All volumes and weights of combustion air and flue gases are given for the 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 No. 415/2012 Coll. (Decree of the permissible level of pollution and its detection and implementation of certain other provisions of Law on air protection), 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 measured parameters of the examined samples of the sludge and meat and bones meal. For the evaluation of the measured values, basic statistical methods were used.

RESULTS AND DISCUSSION

Process parameters of hydrothermal carbonization (HTC) (Fig. 1) confirm the effect of increas-

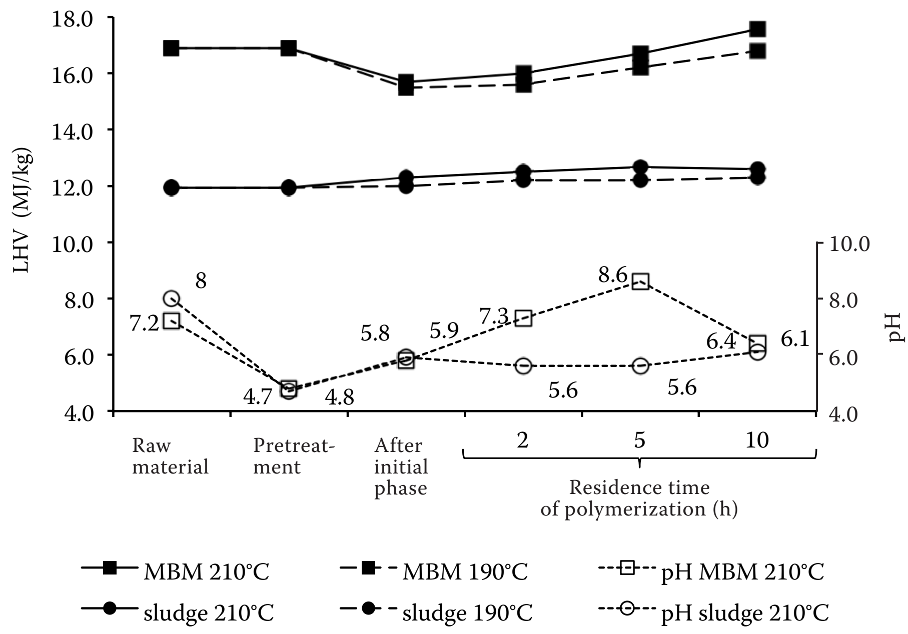

Fig. 1. Parameters of the hydrothermal carbonization of sewage sludge and MBM on processing time
MBM – meat and bone meal
LHV – lower heating value

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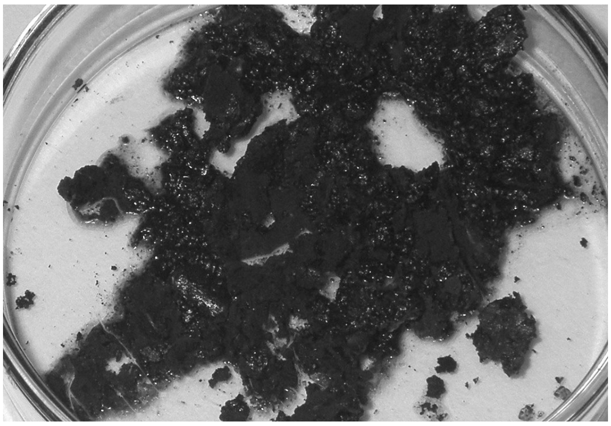

Fig. 2. Meat and bone meal as biochar from hydrothermal carbonization (200°C, 10 h)

ing 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. However, already after the initial phase, there was a substantial growth in relation to the peaks of the LHV. Neither calorific values of MBM, nor calorific values of digested stabilized sludge showed a significant increase after undergoing processing.

The resulting values of elemental analysis are shown for MBM (HTC 200°C, 10 h) (final product shown in Fig. 2) in Table 2. For digested stabilized sludge (HTC 200°C, 4 h) (final product shown in Fig. 3) in Table 3.

The most important results of the elemental analysis were, in terms of energy use and emission concentrations, the content levels of ash, sulphur, chlorine and nitrogen in the examined samples. Especially important for raw MBM is obviously a high concentration of nitrogen (reduced by 47%) and sulphur (reduced by 18% in the solid component of the product) after a reaction time of 2 h or more. This amount was dissolved in gaseous and liquid component. The similar effect was not observed for the sludge, although it is recorded in work of (He et al. 2013). Another important element is chlorine, which is transferred during combustion largely to a gaseous state. Higher concentration is also observed in the sample of MBM. The importance of chlorine lies on one hand in the emissions of HCl – in their 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).

The majority of sulphur similarly goes 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 mean any problem with regards to limit values, as was proved by examined samples. The decisive factor for sulphur concentration in biofuels may be its corrosive behaviour. Other results of elemental analysis meet the optimal parameters for using those biofuel samples in combustion apparatus.

The most important factor for the thermal utilization of solid biofuels is water and ash content in the fuel. The content of water in the sample of stabilized sewage sludge before treatment was high, which subsequently affected fuel heating value. For processed samples the amount of all water was very low, which resulted in a positive contribution to the LHV.

The amount of ash in the examined samples significantly restricts consequential energy use.

Table 2. The resulting values of elemental analysis for meat and bone meals (% wt.)

Sample of meat and bone meal	Sample before treatment		Biochar (200°C, 10 h)	
Water (% wt.)	0.00	3.00	0.00	1.95
Ash (% wt.)	15.49	15.03	44.07	43.21
Carbon (% wt.)	31.11	30.18	38.88	38.13
Hydrogen (% wt.)	8.00	7.76	5.58	5.48
Nitrogen (% wt.)	6.38	6.19	2.97	2.92
Sulphur (% wt.)	0.46	0.45	0.37	0.37
Oxygen (% wt.)	38.56	37.39	7.99	7.84
Chlorine (% wt.)	–	–	–	0.10
HHV (MJ/kg)	–	18.15	–	18.47
LHV (MJ/kg)	16.88	16.38	17.57	17.22

HHV – higher heating value, LHV – lower heating value

doi: 10.17221/59/2013-RAE

Table 3. Elemental analysis for digested stabilized sludge

Sample of digested stabilized sludge	Sample before treatment		Biochar (200°C, 4 h)	
Water (% wt.)	0.00	71.12	0.00	4.18
Ash (% wt.)	49.23	12.18	42.97	41.18
Carbon (% wt.)	11.90	10.31	31.57	30.25
Hydrogen (% wt.)	3.20	3.22	3.95	3.79
Nitrogen (% wt.)	1.40	1.21	2.40	2.30
Sulphur (% wt.)	0.21	0.43	0.79	0.76
Oxygen (% wt.)	32.63	1.49	18.25	17.49
Chlorine (% wt.)	–	0.04	–	0.05
HHV (MJ/kg)	–	4.34	–	13.07
LHV (MJ/kg)	11.93	2.25	12.67	12.14

HHV – higher heating value, LHV – lower heating value

Fig. 3. Digested stabilized sludge as biochar from hydrothermal carbonization (200°C, 4 h)

Biochar of the MBM, and both the sample of raw sewage sludge and its biochar have a high ash content, which exceeds concentrations above 40% wt. The amount of ash significantly affects the thermal properties of examined solid samples and subsequently affects both the selection and the adjustment of combustion equipment.

The most important parameter is the total LHV of the samples before and after the treatment. For samples of MBM there was a slight increase of LHV. The cause of the slight increase of LHV during the processing is a large depletion of oxygen in the fuel at the expense of an increased proportion of ash. The LHV of the stabilized sewage sludge containing 70% wt. of all water is very low. According to the results of the research works of Hartman et al. (2003 and 2005) from the Institute of Chemical Process Fundamentals of the Academy of Sciences of the Czech Republic, which dealt with chemical and fuel characteristics of anaerobically stabilized sewage sludge and its ash, the LHV of dry digested stabilized sludge is 10–11 MJ/kg and it corresponds to the LHV of energy brown coal. The reason for increased LHV at the hydrothermal treatment is a large reduction in all water and oxygen in the sample.

The resulting values of the stoichiometric analysis indicate good thermal emission parameters of examined samples 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 combustion equipment. The concentration of elements in the examined samples, as confirmed by analysis of the samples, is relatively broad. First of all, the amount of oxygen in the raw MBM and the amount of water in raw stabilized sewage sludge define different combustion conditions than for the processed samples of biochar.

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 regard 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.

doi: 10.17221/59/2013-RAE

Table 4. Selected values of stoichiometric analysis

Sample	Unit	Sample before treatment		Biochar	
		meat and bone meal	stabilized sludge	meat and bone meal	stabilized sludge
Theoretical amount of air	(kg/kg)	4.55	2.25	5.95	4.06
	(m^3/kg)	3.50	1.73	4.59	3.13
Theoretical amount of dry flue gases	(kg/kg)	6.61	4.09	7.92	6.21
	(m^3/kg)	3.34	1.56	4.31	3.03
Theoretical concentration of carbon dioxide in dry flue gases	(% wt.)	16.74	9.24	17.65	17.87
	(% vol.)	16.73	12.29	16.40	18.53
Dry flue gases at coefficient of excess air 2.10					
Carbon	(kg/kg)	1.11	0.38	1.40	1.11
	(m^3/kg)	0.56	0.19	0.71	0.56
Sulphur dioxide	(kg/kg)	0.01	0.01	0.01	0.02
	(m^3/kg)	0.00	0.00	0.00	0.01
Water vapour	(kg/kg)	1.11	0.19	1.01	0.72
	(m^3/kg)	1.19	1.39	1.02	0.74
Nitrogen	(kg/kg)	7.28	3.58	9.46	6.46
	(m^3/kg)	5.79	2.85	7.54	5.15
Oxygen	(kg/kg)	1.16	0.57	1.52	1.04
	(m^3/kg)	0.81	0.40	1.06	0.72

CONCLUSION

The work deals with a topical issue of energy utilization of stabilized sewage sludge and other by-products such as meat and bone meal. The determination of process parameters of HTC technology and the subsequent evaluation of the final properties of the samples are the basis for future energy use of examined materials.

Anaerobically stabilized sewage sludge and MBM were processed by HTC to biochar. The effect of reaction temperature on the LHV of the final product is significantly higher than that of residence time. Biomass with high moisture and low energy content, like the selected sludge, is not a suitable raw material to burn or for long transport. HTC is a suitable method both for dewatering material and gaining more valuable product. Because the heating value of biochar largely depends on the input material, it is necessary in terms of combustion to mix examined sludge with another raw material to obtain a final product with a higher calorific value. Under current Czech regulation No. 415/2012, LHV of solid biofuels over 13 MJ/kg is required for combustion apparatus with a nominal input power over 0.3 MW. This requirement is fulfilled by samples of MBM. The biochar of stabilized sludge did not meet this requirement (12.67 MJ/kg). Units of HTC can be a suitable component for a wastewater treatment plant related to its anaerobic stage. This would eliminate the requirements for energy-consuming sanitation of sludge and also problems with the application of sludge compost on the market.

Another requirement for combustion is 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, it 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 both by biochar of the MBM (43.21% wt.) and biochar of the sludge (41.18% wt.). The concentration of chlorine meets the requirements of the regulation No. 415/2012.

doi: 10.17221/59/2013-RAE

Based on the obtained results, it is questionable whether MBM processed into biochar is a good thing. For the stabilized wastewater sludge the HTC technology is one of the effective processing methods for subsequent energy use.

The results show that the main factor affecting LHV of the fuel sample is the final amount of ash. For further energy use of examined material it is recommended to mix another ingredient of biofuel to achieve an improved calorific value to the proportional composition of mixed fuel. This improving factor is affected by the availability of raw materials for blending.

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Received for publication August 26, 2013
Accepted after corrections December 10, 2013

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