Assessment of the Thermal Characteristics of Pellets Made of Agricultural Crop Residues Mixed with Wood

Vasiliki Kamperidou,[a,*] Charalampos Lykidis,[b] and Panagiotis Barmpoutis [c]

The use of agricultural biomass to produce biofuels and energy can provide many environmental and socio-economic benefits. This research project examines the possibility of replacing part of the wood material in a pellet with various proportions of residues of agricultural crops such as medic, maize, wheat bran, wheat straw, sunflower, and cardoon. Such substitution would contribute to the recycling of materials and the sustainable use of wood and other natural resources. It would reduce emissions of gaseous pollutants by replacing the use of other fossil fuels with solid biofuels. The chosen agricultural species, as well as the beech wood used in this work, are among the most widely-available raw materials in Greece and Europe. Specifically, the higher heating value (HHV) of these materials, both separately and mixed, and their respective ash contents (%), a feature highly crucial for their future utilization as biofuels, were estimated and compared among species. Additionally, various mixing ratios of these materials were examined to determine the most appropriate pellet-type biofuels that meet the requirements of the corresponding international standards that pose restrictions on thermal efficiency and ash content.

Keywords: Ash, Biomass; Heating value; Pellets; Residues; Thermal efficiency

*Contact information: a: Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment, Laboratory of Wood Products and Furniture Technology, 54124, Thessaloniki, Greece; b: Institute of Mediterranean Forest Ecosystems and Forest Products Technology, Laboratory of Wood Anatomy and Technology, Terma Alkmanos, Ilisia, 11528, Athens, Greece; c: Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment Laboratory of Forest Informatics 54124, Thessaloniki, Greece; *Corresponding author: vkamperi@for.auth.gr*

INTRODUCTION

Energy production is a profoundly important issue, as it has become central for people's existence and evolution. Since the Industrial Revolution, most of the energy being produced today comes from non-renewable sources that adversely affect the atmosphere and environment while depleting mineral raw material sources. This fact, in combination with national commitments to the European Union regarding the use of biofuels (Directive 2003/30/EC), the substitution of part of the electricity production by renewable energy sources and biomass (Directive 2001/77/EC), and the alignment of the country with the obligations of the Kyoto Protocol, intensifies the significance for research aimed at developing sustainable solutions for the production and use of biofuels.

Moreover, using biomass for energy production contributes many new employment opportunities to rural and forest communities and reduces SO_2 and CO_2 emissions (Telmo and Lousada 2011). There are still some difficulties in using biomass, such as the dispersal and seasonal production of many categories of biomass; this complicates efforts to provide a continuous supply of energy (Cloud 2009). Therefore, a rational design and profound knowledge of its characteristics as a fuel may be required to achieve effective utilization of biomass in energy production.

Compared to other fuel sources, wood is 99% flammable when dry and is the most affordable source of energy (Hannum 2009). The flammability of wood depends on various factors, such as the wood species, moisture content, temperature, dimensions, *etc*. The presence of moisture tends to reduce the calorific value, while the density and the presence of extracts (resin) and lignin increase it (Khider and Elsaki 2012). The bark usually has a higher ash content than the wood, and this may be associated with a lower calorific value (Filbakk *et al*. 2011). Also, the calorific value varies among forest species, and even within the same species (Nurmi 1993). Therefore, the investigation of the thermal characteristics of different species seems to be of paramount importance to understand their behavior and proper use as biofuels.

Pellets are a standardized product characterized by a high consistency, low moisture content (< 10%) (ISO 18134), up to 2% ash content (ISO 18122- domestic), and high density (> 650 kg/m^3), which allows for combustion and high heating efficiency (4063 kcal/kg to 4541 kcal/kg) (Wu *et al*. 2005). In addition, the confined dimensions of the pellets allow for easy storage and handling.

The biomass obtained from agricultural and energy crop residues could contribute to the production of energy in a renewable and environmentally friendly way (Haberl and Geissler 2000). After harvest, copious amounts of lignocellulosic residues remain in the field, creating several problems such as difficulties in movement of the machines in the field and the accumulation of residues material that poses a risk of fire or microorganisms development. Considering that Greece spends great amounts of money (18.28 billion € in 2012, 9.82 billion € in 2016) annually on imports of fuels (Hellenic Statistical Authority 2017) and that approximately 5 million tonnes of lignocellulosic wastes produced annually remain unexploited, there is an opportunity for research to be more focused on the suitability of these remains as alternatives to wood as the raw material to produce solid biofuels. Extensive research has been conducted to exploit these remains in energy production, but the potential of mixing these biomass residues with wood in the production of solid pelletized biofuels has not been thoroughly investigated so far.

Specifically, Dimitrakopoulos and Panov (2001), who studied the use of 13 dominant Mediterranean species as biofuels quantifying their chemical and physical pyric properties, recorded that heating values ranged from 4438 kcal/kg to 5633 kcal/kg. Barboutis and Lykidis (2014) examined the higher heating value and ash content of wood and bark of five Mediterranean evergreen hardwoods and two deciduous species grown in northern Greece. They reported that all species could be used as raw materials for the production of pellets for non-industrial heating purposes, since they meet the related threshold values for all three pellet quality classes. On the other hand, even though they all provided adequate heating values, none of the tested bark samples showed ash content values lower than 3%, meaning that bark can only be carefully used in adequate mixtures with wood. Dahl and Obernberger (2004) investigated the perennial crops giant reed, switchgrass, miscanthus, and cardoon in laboratory scale and pilot-scale combustion test runs, revealing that significantly higher emissions of particulates as well as of gaseous emissions of HCl, SO_2, and NOX were observed from these species, compared to wood pellets. These fuels presented high ash contents with high concentrations of Si, K, and Ca (of low temperature melting ash point) as well as high concentrations of Cl, S, and N, resulting in increased gaseous emissions and problems with the combustion equipment. The solution of blending the perennial crops with wood was proposed, in order to decrease gaseous and particles emissions. Zanetti *et al.* (2017) state that vineyard based pellets are unsuitable for use in traditional pellet stoves, since they did not meet the type B quality standards required for non-industrial use of wood pellet mainly because of the high ash content (>2%) and the high amount of copper (>10 ppm). Carroll and Finnan (2012) mention that compared to wood, willow, or miscanthus, the straws (*i.e.* wheat, barley, and rape) had higher levels of chlorine, sulphur, and nitrogen, compared to wood, which could lead to problems in terms of boiler degradation and gaseous emissions. The cited authors proposed pellet quality improvement through addition of binders and mixtures, developments in boiler design, and update of standards. Gravalos *et al.* (2010) concluded that pellets from forest residues show higher calorific values, lower ash content, and significant higher bulk density compared to pellets from agricultural crop and forage residues. Also, the higher calorific value was produced by ground material from cotton residues and the lowest calorific values was presented by cardoon.

In the present study, an attempt is made to assess the thermal characteristics, such as the ash content and higher heating value (HHV) of pellets derived from agricultural crop residues (medic, maize, wheat bran, wheat straw, sunflower, and cardoon) mixed with beech wood, a raw material readily available in Greece. The utilization of these crop residues and logging or wood processing residues, which have remained unexploited until now, is expected to ensure high energy efficiency in the production of compacted biomass units (pellets), with minimum environmental impact, saving large amounts of biomass and raw materials, while contributing to the long-term reduction of gaseous pollutant emissions and particulate matter (PM), the achievement of a more sustainable forest management, and the conservation of ecosystems.

EXPERIMENTAL

Materials

For this experiment, samples of the residues of the most important agricultural plants cultivated in Greece were collected, including medic (*Medicago sativa* L.), maize (*Zea mays*– hybrid of Pioneer 514), wheat bran (*Triticum aestivum*), wheat straw, cardoon (*Cynara cardunculus*), and sunflower (*Helianthus annuus* L.). Wheat and maize residues were chosen, since these species account for a high share of the cereals produced in the EU-28 (86%, with spelt and barley 2007-15), while medic, cardoon, and sunflower are classified among the most common and significant energy crops (Eurostat 2016).

For maize, seeds and crop residues were harvested separately, while for sunflower, the raw material was obtained from residues arising from the seed harvest (head). Additionally, wood and bark samples at various heights of three beech (*Fagus sylvatica* L.) logs were collected. All species were obtained from regions of Central Macedonia and Thrace (Greece) and were placed in a conditioned room (20 °C ± 2 °C, 60% ± 5% relative humidity) to attain a nominal equilibrium moisture content until steady mass was achieved.

Methods

All materials were ground *via* a portable chipper. The bulk samples were reduced by coning and quartering to a representative sample of approximately 0.5 kg. The samples were subsequently air-dried and ground using a rotating-blade Wiley mill (Thomas Scientific, Swedesboro, USA) with a 0.7-mm sieve.

The determination of ash was implemented following the methodology described in EN 14775 (2004). The samples with mass of at least 1 g were weighed to the nearest 0.1 mg in the pre-weighed porcelain crucibles and transferred in a cold muffle furnace (WMW AG, Heraeus MR 170, Leipzig, Germany) with a ventilation rate of about 5 changes per min. The samples were then heated to 250 °C within 50 min, and the temperature was kept constant for 60 min, to allow the volatiles to leave the sample before ignition. In the next step, the temperature was increased to 550 °C within 60 min and was maintained at that level for 3 h. In sequence, the crucibles were transferred to an empty desiccator without a lid for 5 min followed by 15 min with a closed lid and then weighed. To ensure complete incineration, the samples were reloaded in the hot furnace for 30 min intervals and were reweighed according to the above procedure until the mass changes were lower than 0.2 mg. The ash content on a dry basis was calculated according to Eq. 1,

$$A_d\ (\%) = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \qquad (1)$$

where A_d is the ash content (%), m_1 is the mass of the empty crucible (g), m_2 is the mass of the crucible plus the dried test sample (g), and m_3 is the mass of the crucible plus ash (g).

The ash measurements were performed in 3 replicates for each material. The moisture content was determined according to the process of EN 14774-3 (2009).

HHV corresponds to the absolute value (expressing distance from 0) of the specific energy of combustion in calories per unit mass of solid biofuel burned in oxygen (in a calorimetric bomb) under specified conditions. The HHV was determined in a isoperibol bomb calorimeter (Parr 1261, Parr Instrument Company, Moline, IL, USA) according to the method described in EN 14918 (2009). Sample pellets with mass of 1.0 g ± 0.1 g, with a diameter of 13 mm were produced in the shape of pills using a hydraulic pellet press (Custom made, Athens, Greece) applying a load of approximately 7000 kg for 1 min. The pellets were weighed to the nearest 0.01 g in a crucible and then placed inside a Paar 1108 oxygen combustion bomb in contact with 10 cm of pre-weighed platinum ignition wire. The bomb was subsequently charged with oxygen (purity of 99.7%) at 30 bar ± 2 bar and submerged in a stainless steel bucket containing 2000.0 mL of distilled water. Prior to filling the bucket, the water was conditioned in a water bath at 33 °C ± 0.5 °C. The calorimeter jacket was maintained at a constant temperature by circulating the water at 35 °C to maintain a slightly higher temperature than the final temperature of the calorimeter and to assure that evaporation losses were minimized. The HHV measurements were conducted in 6 replicates for each material. Prior to starting the above measurements, the calorimeter was calibrated and validated with 6 individual calibration runs using benzoic acid pellets. The HHV values were expressed in kcal/kg. Sulphur and chlorine adjustments were not conducted because they are present in low concentrations in wood fuels (Lehtikangas 2001).

Afterwards, a theoretical estimation of the fuel characteristics of the pellets produced from the selected species was implemented. This was based on the bark and wood ash content as well as the calorific values that were determined, considering the various barked stem diameters of the raw materials. Hence, the following equations were used (Eqs. 2 and 3),

$$ASH = a_1 \frac{Z}{100} + a_2 \frac{100 - Z}{100} \qquad (2)$$

where ASH is the total ash content (%), Z is the bark percentage (%), a_1 is the ash content of bark (%), and a_2 is the ash content of wood (%),

$$HHV = b_1 \frac{Z}{100} + b_2 \frac{100-Z}{100} \quad (3)$$

where HHV is the total HHV (kcal/kg), b_1 is the HHV of bark (kcal/kg), and b_2 is the HHV of wood (kcal/kg).

To process results and for the statistical analysis of mean values, the statistical package SPSS Statistics (IBM SPSS, Version SPSS 22, New York, USA) (ANOVA - a = 0.95, Least Significant Difference; LSD) was used, while the comparison ratios were implemented using the MATLAB program (MathWorks, Version Matlab 2016a, Natick, MA, USA).

RESULTS AND DISCUSSION

According to the results presented in Table 1, the lowest achieved ash content was that of beech wood (0.5%), while the bark of beech, as expected, had a higher ash content (7.73%). The highest ash content among the species examined in this study was recorded by wheat straw (9.9%). Maize, whether referred to the seed or the crop residues, as well sunflower showed relatively low ash contents, even though they are higher than the allowed limits of the standard requirements for non-industrial purposes (residential uses, such as heating, cooking, *etc*.) (>2.0) (ENplus Handbook, Part 3: Pellet Quality Requirements, European Pellet Council 2015).

Regarding the calorific value generated from the examined species, sunflower had the highest value (4900 kcal/kg), while wheat bran marked the lowest (3633 kcal/kg). Both the wood and bark of beech exhibited very satisfactory heating efficiency, while the yield of wheat straw and cardoon was slightly lower. Maize residues have a moderate heating value, while wheat, medic, and the seed of maize have lower thermal yields (3633 kcal/kg to 3872 kcal/kg). The moisture content of the raw materials fluctuated at low levels, from 7.4% to 10.1%.

Table 1. Ash Content, Calorific Value, and Moisture Content of the Studied Raw Materials (Wood and Energy Crop Residues)

Properties		Beech		Medic	Maize (seed)	Maize (resid.)	Wheat Bran	Wheat Straw	Cardoon	Sunflower
		Wood	Bark							
Ash (%)	MO[a]	0.50	7.73	6.1	3.10	2.6	6.8	9.9	5.2	3.7
	SD[b]	0.07	0.53	0.10	0.00	0.17	0.31	0.36	0.42	0.55
	CV[c]	14.4	6.9	0.6	13.2	1.0	2.0	1.9	8.0	15
	N[d]	3	3	3	3	3	3	3	3	3
HHV (kcal /kg)	MO	4589.9	4442.6	3656.8	3871.9	4039.2	3632.9	4373.8	4294.9	4899.6
	SD	9.62	1.9	23.9	29.15	26.29	38.24	262.9	152.96	95.6
	CV	0.2	0.0	0.6	0.3	4.2	2.3	6.0	3.6	1.9
	N	6	6	3	3	3	3	3	3	3
MC (%)	MO	7.9	9.5	9.8	8.6	10.1	9.5	7.4	8.9	10.1

Note: a: mean value, b: standard deviation, c: coefficient of variation, and d: number of specimens

The ash content is perhaps the most crucial qualitative characteristic of a biomass material, since it is closely related to problems of ash removal, corrosion of equipment, slagging tendency, and deposit formation in the furnace. According to the specifications of standards for biofuels, restrictions are posed on the uses and utilization of various materials depending on their ash contents (Dahl and Obernberger 2004).

According to Table 2, by maintaining a constant share of beech wood at 25% and using various blends of the other species examined in this research for the remaining 75%, no combination can be utilized in pellets of domestic use (up to 2% ash content). The cases of mixing pure wood or bark-containing beech wood with maize, referring to either seeds or residues, revealed the production of pellets that can be consumed for industrial use (in plants, greenhouses, barns, stalls, *etc*.) (up to 3%), as well as the case of mixing pure beech wood with sunflower. Mixing beech wood of larger diameter (for example, 20 cm) with sunflower could also yield material of acceptable ash results (European Pellet Council 2015).

Table 2. Estimation of the Ash Content of Pellets Maintaining a Constant Share of Beech Wood of 25% Mixed with Various Types of Energy Crop Residues

Beech Share 25%		75%						
		Medic	Maize (seed)	Maize (residual)	Wheat Bran	Wheat Straw	Cardoon	Sunflower
Pure Wood		4.70	2.45	2.07	5.22	7.55	4.02	2.90
Barked Wood	*R** = 5	4.94	2.69	2.31	5.46	7.79	4.26	3.14
	R = 7.5	4.86	2.61	2.23	5.38	7.71	4.18	3.06
	R = 10	4.82	2.57	2.19	5.34	7.67	4.14	3.02

R: radius (cm)

When maintaining a constant beech wood share at 50% combined separately with the other species examined in the present research (Table 3), some combinations gave pellets with ash content levels that correspond to residential use. Such cases include mixing pure wood with maize seeds or maize residues, as well as mixing bark-containing wood (irrespective of diameter) with maize residues. The 50:50 ratio (wood:energy plant) also gave many combinations for pellets suitable for industrial use in addition to those for residential use, such as the mixtures of pure wood with cardoon and the mixture of pure or bark-containing wood with sunflower. The involvement of maize seeds in pellet production is particularly important, as it contributes to the increase of pellet cohesiveness, which may be attributed to the starch it contains. In contrast, maize residues, although characterized by lower ash contents and higher calorific yields, do not contain the same substances that contribute to the consistency of the pellets.

Table 3. Estimation of the Ash Content of Pellets with a Constant Share of Beech Wood of 50% Mixed with Various Energy Crop Residues

Beech Share 50%		50%						
		Medic	Maize (seed)	Maize (residue)	Wheat Bran	Wheat Straw	Cardoon	Sunflower
Pure Wood		3.30	1.80	1.55	3.65	5.20	2.85	2.10
Barked Wood	*R** = 5	3.78	2.28	2.03	4.13	5.68	3.33	2.58
	R = 7.5	3.62	2.12	1.87	3.97	5.52	3.17	2.42
	R = 10	3.54	2.04	1.79	3.89	5.44	3.09	2.34

R: radius (cm)

In Table 4, different mixing ratios were examined involving pure or bark-containing beech wood (50% or 60%, of diameter 15 or 20 cm) in combination with maize seeds, whose presence is essential to the increase the cohesiveness of pellets, as well as maize residues, which presented the lowest ash content, and sunflower residues, which had the highest calorific value, to identify the optimal proportions of biomass materials that could ensure the production of pellets of high level qualitative characteristics according to the standards (European Pellet Council 2015).

Table 4. Various Proportions of Beech Wood in Mixture with Different Types of Energy Crop Residues into Pellets, with the Lowest Possible Ash Content and Maximum Heating Value

Pure Beech Wood	Bark-containing Beech Wood *R* = 7.5	Bark-containing Beech Wood *R* = 10	Maize (seed)	Maize (residual)	Sun-flower	Ash (%)	HHV (kcal/kg)
50%			10%	40%		1.60	4297.8
	50%		10%	40%		1.93	4290.7
		50%	10%	40%		1.84	4292.3
60%			10%	30%		1.39	4352.9
	60%		10%	30%		1.78	4344.3
		60%	10%	30%		1.68	4346.3
50%			10%		40%	2.04	4642.0
	50%		10%		40%	2.37	4634.8
		50%	10%		40%	2.29	4636.5
60%			10%		30%	1.72	4611.0
	60%		10%		30%	2.11	4602.4
		60%	10%		30%	2.01	4604.4

According to the results (Table 4), all of the proportions examined resulted in biomass pellets that met the requirements of the standard regarding ash content for industrial use. Additionally, most categories fulfilled the requirements of the standards for residential use (class EN plus B, European Pellet Council 2015), except for the mixtures of 50% bark-containing beech wood (of radius 7.5 or 10) with 10% of maize seed and 40% of sunflower, as well as the mixture of 60% bark-containing beech wood (of 7.5 radius) with 10% of maize seeds and 30% of sunflower, which were the cases of the highest ash content among all mixture ratios. These values of high ash content could be attributed to the presence of beech bark, as well as the higher lipid content of maize seeds and the sunflower residues (coming from residues arising from the seed harvest). All of the ratios examined in this research work presented satisfactory calorific yields, with the highest heating values being recorded in the cases when the maize residues are replaced by sunflower residues.

CONCLUSIONS

This research work focused on the accomplishment of the improvement of the utilization of remaining agricultural crop residues in pellets production, replacing a part of wood raw material of the solid biofuel. The sustainable use of wood and the reduction of cost of raw materials intended for pellets production motivated this work.

According to the findings, part of the wood material intended for the production of solid biofuel (pellets) could be successfully saved and be substituted by residues from the agricultural crops of medic, maize, wheat bran, wheat straw, sunflower, and cardoon mixed in appropriate proportions with beech wood. Beech, which is a raw material readily available in Greece and whole Europe, proved to be suitable for pellets production, since it recorded the lowest ash content and the highest calorific efficiency among all the materials used here. Maize and sunflower presented relatively low ash contents, but they could not fulfill the standard requirements for non-industrial purposes (>2.0), as well as the wheat straw that marked the highest ash content among the species. Sunflower exhibited the highest calorific value, classifying the species as suitable to be included in the raw material in pellet production, while wheat bran marked the lowest heating efficiency and could be included only under the term of limited participation.

Mixtures of pure or bark-containing beech wood (50% or 60%, using logs of 15 or 20 cm diameter), together with maize seeds (which contribute to the accomplishment of improved pellets consistency), maize residues (that demonstrated one of the lowest ash contents), and sunflower, (which presented the highest calorific value), could contribute to the production of pellets with optimum thermal characteristics according to the current standards, recording lower ash content and, at the same time, higher thermal efficiency values.

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