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ARTICLE

Thermal analysis of aboveground biomass of the two species cultivated in artificial forest plantations in marginal lands of Ukraine

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ABSTRACT
The thermal degradation of the wood and bark of the Black locust (*Robinia pseudoacacia L.*) and Scots pine (*Pinus sylvestris L.*) cultivated as monocultures in marginal lands of Ukraine has been investigated using thermogravimetric methods of differential thermal analysis. The activation energy of the trees' bark and wood destruction processes were calculated for each temperature range. Comparison of DTA curves showed that the thermal effect of oxidative destruction of the Black locust bark as a whole is greater than that of wood, which correlates with a decrease in the activation energy.

KEYWORDS
Scots pine; Black locust; thermal degradation

Introduction

One response to the challenges of climate change and energy supply is the cultivation of woody plants in marginal lands for biofuels [1]. This means that the poorest land must be operated under given price, cost and other conditions [2]. Recently, most articles available on biofuels take a technical approach, focused on energy security or climate change issues [3–5]. In 2017, the energy intensity of Ukraine was 0.27 tonnes of oil equivalent (toe) for $1000 of GDP compared to EU countries, where the indicator ranged from 0.08 to 0.13 [6]. Ukraine's sustainable development strategy includes creation of its own energy base, using regional renewable energy sources for energy security. The artificial forest plantations are subdivided into industrial and protective plantations, plantations of various technical crops, short-rotation technical biomass production plantations, etc., [3]. The Black locust and Scots pine are suitable tree species for establishing energy plantations in marginal lands [7,8]. Currently, logging residuals are the main source of the biomass consumed in the growing number of Ukraine's modern district heating plants in cities and municipalities [9]. Meantime, the share of wood chips from the clearing of naturally over-grown abandoned lands is increasing. The thermal

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decomposition of wood residues is a subject of significant recent research [10–12]. The thermal degradation of plant polymers attracts increasing attention as a promising way training valuable organic products and, accordingly, energy [13–15]. This interest includes to use the woody plant biomass for energy sources and goods [16,17]. The increasing importance of biomass as a renewable energy source has led to a need for reliable and detailed information [18,19]. Physical and chemical properties of timber, such as density, heat capacity, thermal conductivity, moisture content, the ratio of the main biopolymer components, physical and chemical features of hemicellulose, cellulose and lignin content and the quality of extractives determine the parameters of thermal destruction [20,21].

The best strategy for genetic improvement of Scots pine juvenile wood for bioenergy production is to decrease and stabilise the content of extractives among trees and then focus on increasing the cellulose: lignin ratio [22]. The main questions concern the composition of structural components of aboveground biomass of plants. The carbohydrate complex of wood is holocellulose whose composition includes cellulose and hemicelluloses. Hemicelluloses, in turn, are represented by pentosans, hexosans, and uronic acids, which contain carboxyl, acetyl groups and methoxyls. The output of holocellulose for deciduous wood, as Black locust, varies in the range of 72–79%, lignin – 18–22%. Wood coniferous species contain 48–50% cellulose, 28–30% lignin, and 23–26% hemicellulose. Hemicellulose coniferous species contain galactoglucomannan and arabinoglucuronoxylan [23].

Black locust (*Robinia pseudoacacia* L.) and Scots pine (*Pinus sylvestris* L) are dominant woody species in the northern part of Ukraine's steppe zone. The main goal of this research is to analyse the processes of thermal destruction of wood and bark of the main forest-forming species cultivated in the marginal lands.

Materials and methods

The study was conducted in different parts of Dnipropetrovsk region (47–49°N; 33–37°E) located in the Northern Steppe of Ukraine and covers 31,974 km^2. The climate is temperate continental with a mild winter having a small amount of snow, and a hot and dry summer with strong southern winds. The average annual temperature (during the last 25 years) is 10.6°C, the total precipitation is 400–490 mm.

Forest plantations of forestry enterprises of the Northern Steppe of Ukraine have no significant wood resources. Felling was carried out only for the formation and rehabilitation of forests. The volume of exchangeable and commercial timber is designed for sanitary felling, taking into account the actual condition of plantations. Forestry measures include care pruning, restorative, selective and continuous sanitary felling (Table 1).

The average annual harvest of commercial timber in forestry is 34.3 thousand m^3, including lumber and round billets – 2.5 thousand m^3, firewood for technological needs – 9.4 thousand m^3, firewood for heating – 21.7 thousand m^3. The survey was conducted in forest plantations with Black locust and Scots pine within the responsibility of Dnipropetrovsk Administration of Forest and Hunting Management. The sites were established in monoculture of the Scots pine and Black locust mature stands in forest enterprises.

Table 1. Forestry activities related to forest formation and rehabilitation (per year).

Logging type	Square, ha	Timber harvesting, m^3		
		Stem stock	Exchange stock	Business wood
Selective sanitary	502	7341	6445	236
Total sanitary	266	37,991	30,545	4075
Reforestation	22	1624	1484	388
Lighting	152	180	139	0
Cleaning	15	31	2	0
Thinning	67	1271	1061	120
Check points	386	9233	7801	862

As to the object of the survey, there were components of aboveground biomass (trunk wood and trunk bark) from two Scots pine and Black locust forest plantations created in sandy-loam soil. Various soil and environmental characteristics can be used to define soil marginality [24]. The classification schemes of trophotops and hygrotops based on the two edaphic factors (soil fertility and humidity) are used to estimate some forest sites created in sandy and sandy-loam soils as marginal [25,26]. The creation in the marginal lands' artificial energy plantations consisted of Scots pine and Black locust stands as part of the reserve, recreational and protective forests in line with regional environmental policy.

Samples of wood and bark from Scots pine and Black locust were taken for thermogravimetric analysis, from felled trees in the tested plantation. All timber accumulated through felling in the study area is used as a renewable energy resource. A comparative thermogravimetric analysis of woody plant biomass samples from Black locust and Scots pine was carried out to obtain information about the thermal stability of the wood and bark. The analysis was performed using the derivatograph Q-1500D of the 'F. Paulik-J. Paulik-L. Erdey' system.

Differential mass loss and thermal effects were recorded. The results of the measurements were processed with the software package supplied with the device. Samples of wood and bark biomass were analysed dynamically at the heating rate of 10°C/min in the air atmosphere. The weight of samples was 100 mg. Aluminium oxide was used as the reference substance.

The activation energy of thermo-oxidation destruction samples was determined by the method of Broido [27]. The value of the double logarithm for each temperature was calculated using the dependence:

$$\ln\left(\ln\frac{100}{100-\Delta m}\right) = -\frac{E}{R}\cdot\frac{1}{T} \tag{1}$$

Where: m – sample mass, %; E – the activation energy, kJ/mol; R – universal gas constant, 8,314 J/(mol·K); T – temperature, K.

The regression equations and their correlation coefficients were determined by analysing the curves constructed in the coordinates of the Broido equation for different temperature ranges corresponding to the stages of moisture evaporation, volatile organic substances and the stages of decomposition of cellulose, hemicellulose and lignin.

All the results obtained were treated by statistical methods using the StatGraphics Plus 5 software package at the significance level of 0.95% (P value < 0.05).

Results and discussion

The process of thermal destruction of woody plant biomass is mainly related to the structural components that are part of the plant material. Compared to wood, the bark contains more ash, extractive substances and lignin, but significantly less cellulose (almost 3 times) and pentosanes. The content of pentosanes in the bark of coniferous and hardwood species is almost the same [14]. The characteristic feature of the bark is the high content of tannins, as well as the presence of a waxy crust substance – suberin [28]. Obviously, differences in the chemical composition of wood and bark cause the difference in their thermal effects. The average calorific value for Black locust bark is 10% higher than for this species' wood [29]

Thermogravimetry data make possible a comparative analysis of the thermal characteristics of wood and bark of Black locust and Scots pine. Figures 1 and 2 show the TG and DTG data for the wood and barks studied. Figure 3 shows the curves of differential thermal analysis of the two studied trees' wood and bark.

At the first stage, in the temperature range of 20–130°C, the unbound (free) water is removed from the cell cavities, accompanied by the endothermic effect (Figures 3 (a,b)). The water removal from the bark of Scots pine is the slowest with the highest values of E_{act} and endothermic effect (t = 110°C). The mass loss of the samples equals 4.8%. The endothermic minimum of thermal decomposition of the Black locust wood and bark is achieved at a temperature of ~100°C. Meantime, the processes

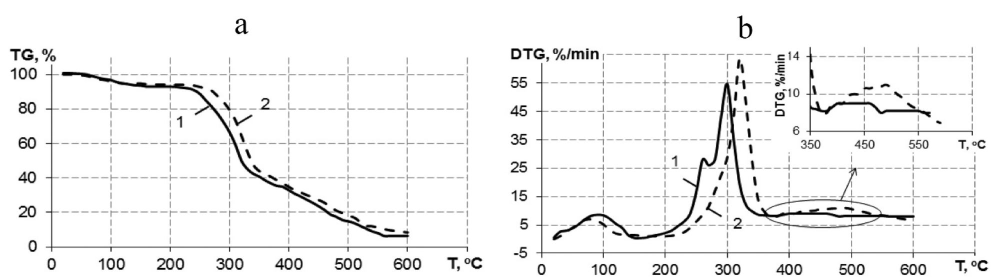

Figure 1. The mass loss (TG) curves (a) and the differential-thermogravimetric (DTG) curves (b) for the wood of Black locust (1) and Scots pine (2) in the oxidising medium.

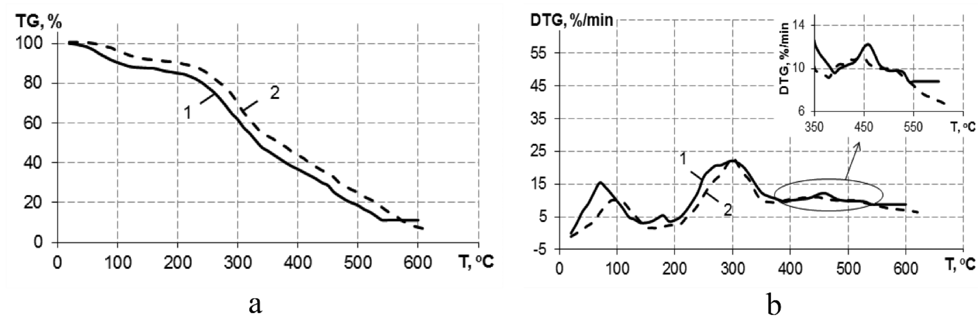

Figure 2. The mass loss (TG) curves (a) and the differential-thermogravimetric (DTG) curves (b) for the bark of Black locust (1) and Scots pine (2) in the oxidising medium.

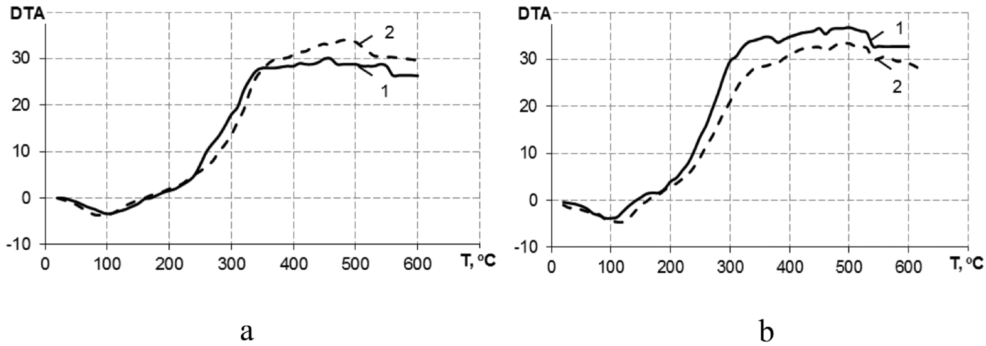

Figure 3. The differential thermal analysis (DTA) curves for the wood (a) and bark (b) of Black locust (1) and Scots pine (2) in the oxidising medium.

proceed with different intensities (Figures 1(b),2(b)), and the samples lose 3.3 and 9.6% of the mass, respectively. This thermal effect explains the high rate of mass loss by the Black locust bark samples. At the same time, 3.2% of the total mass of water is removed from the samples of Scots pine wood with the least endothermic effect at the t = 90°C.

The second stage of the samples' thermal degradation is observed within the 100 (130)–220-(240)°C temperature range. This stage is characterised by the decomposition of organic substances, namely pentosanes, which are the least stable constituent part of hemicellulose, and also by the release of volatile compounds and residual amounts of hygroscopic moisture presenting between fibrils of the cellular wall [14].

The Black locust bark loses 10% of its mass at this stage at the expense of the decomposition of pentosanes which are contained in the bark of hardwoods in larger quantities and which are more susceptible to hydrolysis and dehydration than other wood polysaccharides [30]. The constitutional water removal also occurs with a small endothermic effect at the temperature of 180°C. The remaining samples lose their masses insignificantly, from 2.4 to 5%.

The third stage of thermo-oxidative degradation of the samples proceeds within the 220(240)–340(360)°C temperature range, wherein the final decomposition of the hemicellulose, as well as cellulose and lignin takes place. The thermal degradation of the samples under study is accompanied by high values of the E_{act}, the greatest mass loss of the wood of Black locust (51.71%) and Scots pine (51.20%). The Black locust and Scots pine bark losses are 32.6% and 37.2% of the initial samples mass, respectively. As shown in Figure 4, the logarithmic relationships of Δm depending on temperature make it possible to distinguish four main temperature intervals of thermal destruction of both wood and bark of Black locust and Scots pine.

The wood loss rate curves for both trees have pronounced narrow peaks (Figure 1(b)). The predominant decomposition of cellulose in this range is fixed with a maximum at t = 320°C for the Scots pine wood and t = 300°C for the Black locust wood. Figure 1(b) shows the profiles of the wood mass loss rate curves for both trees. There are narrow peaks for the predominant decomposition of cellulose in this range with a maximum at the t = 320°C for the Scots pine wood and t = 300°C for the Black locust wood. At the same time, the presence of a low-intensity peak at the t = 280°C for the Black locust bark

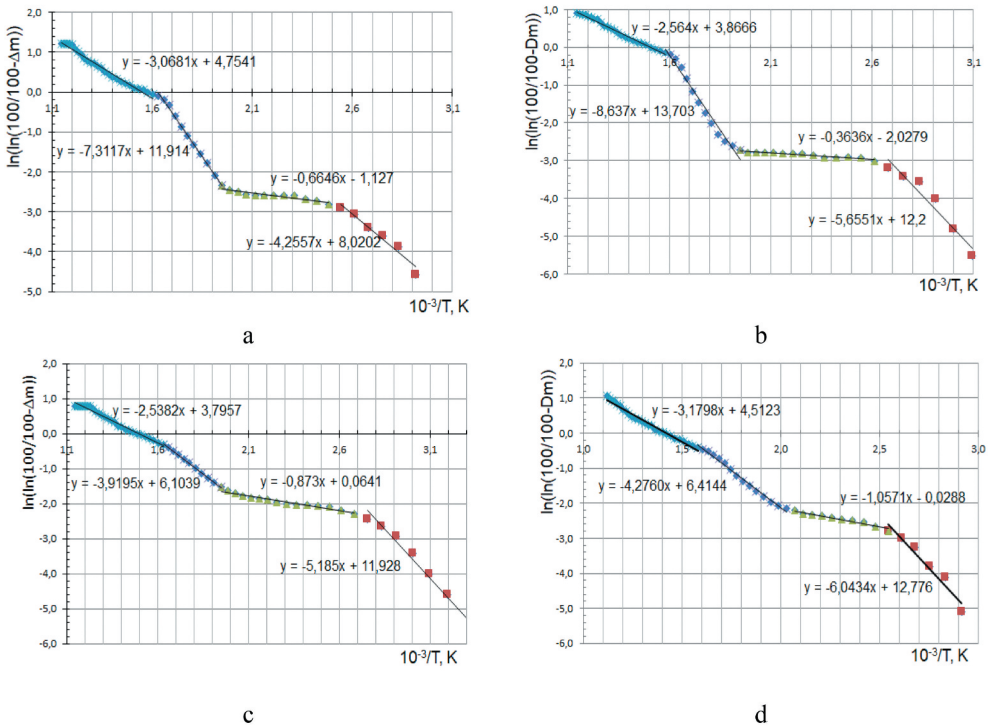

Figure 4. Logarithmic dependence of Δm on T for thermal degradation of the Black locust (a) and Scots pine (b) wood, as well as of the Black locust (c) and Scots pine (d) bark in the oxidising medium.

was fixed in the range of hemicellulose decomposition (Figure 2(b)). The Scots pine bark described process starts at a lower temperature (220°C) because of the lower content of cellulose.

The Scots pine bark described process starts at a lower temperature (220°C) owing to the lower content of cellulose. The mass loss rate curves for the bark of Black locust and Scots pine (Figure 2(b)) have 2.5–2.7 times lower and broader peaks than for the wood, with a less expressed maximum at the t = 300°C. Although the samples degrade at the same speed, the decomposition of the Black locust bark is accompanied by somewhat higher exothermic effect (Figure 2(b)), because of the different chemical compositions of the bark. There is a small plateau on the DTG curve for the Scots pine bark at temperatures of 270–300°C, which is explained by the additional decomposition of hemicellulose (as in the case of the Scots pine wood). As for thermal degradation of the wood, because the two tree species are very similar in chemical composition, their DTA dependence curves are also very similar (Figure 3(a)).

The fourth stage of thermo-oxidative destruction within the temperature range of 340–360–600-620°C is associated with decomposition of the whole biomass organic matter including lignin and the formation of mineralised residues of insignificant mass. Figure 3 shows that exothermic effects overlap on the DTA curves after 360°C. There are minor peaks on the DTG curves in the 390–460°C range, which correspond to the end of lignin decomposition. The highest exothermic effect of thermo-oxidative degradation of Black locust bark samples is observed at a temperature of 200°C. From

350°C the exothermic effect is the smallest. In the samples of Scots pine, the exothermic effect of the bark is higher by 14–41%, than that of the wood in the 200–350°C temperature intervals. Table 2 shows the statistically reliable mathematical models for estimating the dependence of mass loss on the degradation temperature of the above-ground biomass components of the studied samples.

The excessive activation energy of thermos-oxidative degradation is necessary for the destruction of chemical bonds that form the basis of the polymer chain [15]. The values of this parameter increase with the formation of bonds between macromolecules of a polymer. The effective activation energy of thermos-oxidation destruction is a measure of the stability of biopolymers. Accordingly, the greater the degree of cross-linking of macromolecules, the higher is the value of the activation energy.

Analysis of values of activation energy, temperature ranges, and intervals of degree change conversions for biomass of investigation woody plants (Table 3) shows that the heat resistance of the samples is almost the same between the 20 and 130°C. The samples of both species manifest the highest heat-resistant properties in the 130–600°C range. At the same time, the bark of the Black locust is less heat-resistant than that of the Scots pine in almost the entire temperature range studied (20–560°C).

Conclusions

The differences in the chemical composition of Black locust and Scots pine wood and bark in the main biopolymer components cause the uneven kinetics of the thermal destruction of the investigated structural components of the above-ground biomass, as evidenced by the value of the activation energy in the process.

Table 2. Equations at different temperature intervals.

Temperature, °C	Equation	Value of confidence
Wood of Black locust		
70–130	$\Delta m = -4.2557\ T + 8.0202$	0.957
130–240	$\Delta m = -0.6646\ T - 1.1275$	0.848
240–350	$\Delta m = -7.3117\ T + 11.914$	0.997
350–600	$\Delta m = -3.0681\ T + 4.7541$	0.977
70–600	$\Delta m = -3.2624\ T + 4.9056$	0.954
Bark of Black locust		
30–100	$\Delta m = -5.185\ T + 11.928$	0.961
100–240	$\Delta m = -0.873\ T + 0.0641$	0.949
240–340	$\Delta m = -3.9195\ T + 6.1039$	0.999
340–600	$\Delta m = -2.5382\ T + 3.7957$	0.984
30–600	$\Delta m = -2.411\ T + 3.5762$	0.960
Wood of Scots pine		
50–100	$\Delta m = -5.6551\ T + 12.2$	0.939
100–240	$\Delta m = -0.3636\ T - 2.0279$	0.898
240–360	$\Delta m = -8.637\ T + 13.703$	0.982
360–600	$\Delta m = -2.564\ T + 3.8666$	0.993
50–600	$\Delta m = -3.0868 + 4.3599$	0.921
Bark of Scots pine		
70–120	$\Delta m = -6.0434\ T + 12.776$	0.949
120–220	$\Delta m = -1.0571\ T - 0.0288$	0.961
220–360	$\Delta m = -4.2760\ T + 6.4144$	0.991
360–620	$\Delta m = -3.1798\ T + 4.5123$	0.982
70–620	$\Delta m = -2.9112\ T + 4.1207$	0.978

Table 3. Activation energy and degree of decomposition.

Black locust	Wood	T, °C	70–130	130–240	240–350	350–600	70–600
		E_{act}, kJ/mol	35.382	5.525	60.789	25.508	27.123
		α	0 ÷ 0.059	0.059 ÷ 0.096	0.096 ÷ 0.632	0.632 ÷ 1	–
	Bark	T, °C	30–100	100–240	240–340	340–600	30–600
		E_{act}, kJ/mol	43.108	7.258	32.587	21.103	20.045
		α	0 ÷ 0.108	0.108 ÷ 0.221	0.221 ÷ 0.587	0.587 ÷ 1	–
Scots pine	Wood	T, °C	50–100	100–240	240–360	360–600	50–600
		E_{act}, kJ/mol	47.017	3.023	71.808	21.317	25.664
		α	0 ÷ 0.044	0.044 ÷ 0.070	0.070 ÷ 0.629	0.629 ÷ 1	–
	Bark	T, °C	70–120	120–220	220–360	360–620	70–620
		E_{act}, kJ/mol	50.245	8.7888	35.551	26.437	24.204
		α	0 ÷ 0.064	0.064 ÷ 0.116	0.116 ÷ 0.511	0.511 ÷ 1	–

Black locust wood and bark are slightly different in their ability to hold water, as shown by the thermal effects of water loss and temperature peaks on the DTG curve. Black locust wood is characterised by higher thermal stability than bark.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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