

Article

Research on the Kinetics of Pyrolysis of Wood-Based Panels in Terms of Waste Management

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Received: 18 July 2019; Accepted: 26 September 2019; Published: 27 September 2019



Abstract: Currently, there is a lot of interest in implementing the idea of a circular economy along with searching for optimal methods of waste management in terms of raw materials and energy. Waste wood-based floor panels are part of this discussion with regard to its management. The interest in this waste results from statistics and the prediction of its future quantities on the waste market. The separation and testing of individual layers of the waste floor panel was undertaken to answer the following question: Is it reasonable to mechanically separate the contaminated upper panel layer from the remaining part (which is suitable for material recycling) and subject it to thermal transformation methods? Thermogravimetric studies did not confirm the rationale of mechanical separation of layers for further management. Therefore, the use of pyrolysis was proposed as an alternative by showing the advantages of this process in the thermal transformation of the tested waste. The analyzed kinetics of this process included: mass loss, the influence of heating rate on the decomposition process, the impact of volatile parts in the substrate on the rate of mass loss, and the share of coke residue. Empirical formulas of the tested substrates in the molecular formula C–H–O–N (carbon-hydrogen-oxygen-nitrogen) were also proposed to assess its energy usefulness by entering the analyzed waste into a Van Krevelen diagram.

Keywords: kinetics; pyrolysis; wood-based panels; Van KREVELEN systematics; TG-MS/FTIR analysis

1. Introduction

It is estimated that more than 10 million m³ of various types of wood materials are currently produced in Poland, over 3.5 million m³ of which are MDF (medium-density fiberboard) and HDF (high-density fiberboard) [1].

At a time of great emphasis on the recovery of materials according to the idea of a circular economy (CE) and following the hierarchy of waste management (obligatory for business entities) with regard to the post-consumer wood waste, such as floor panels, two options for their management were considered as below [2–5]:

- mechanical and thermal method,
- thermal method.

The proposed methods fit into the idea of a circular economy, whose most important assumptions are presented below. The most popular definition of the circular economy was presented by the Ellen MacArthur Foundation: “A circular economy is one that is restorative and regenerative by design and aims to keep products, components, and materials at their highest utility and value at all times, distinguishing between technical and biological cycles” [6]. Another definition was provided in the publication [7], i.e., a circular economy is “a concept used to describe a zero-waste industrial economy

that profits from two types of material inputs: (1) biological materials are those that can be reintroduced back into the biosphere in a restorative manner without harm or waste (i.e. they breakdown naturally); and, (2) technical materials, which can be continuously re-used without harm or waste" [8]. According to the authors of the publication [9], the circular economy shall be regarded as a "project and business modeling strategy that is, slowing, closing and narrowing the resource loop".

The circular economy in [10] is defined as "a regenerative system in which input resources and waste, emissions and energy leaks are minimized by slowing, closing and narrowing the material and energy loops. This can be achieved through long-term design, maintenance, repair, reuse, regeneration, renewal and recycling."

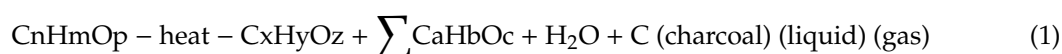
To a large extent, the circular economy in waste management is currently focused on resource recovery, the rational management of non-renewable resources, and environmental impact prevention. In the past, waste management was understood only in terms of storage and incineration [5,11].

The former of the aforesaid methods could include two processes. The first mechanical one involves the roughing (rupture, ...) of the upper layer, which is theoretically more contaminated with chemical impurities—mainly urea–formaldehyde resins—due to its exposure to direct use and requirements on abrasion, waterproofness, etc. This layer would require thermal transformation. The second process is related to the layers lying below the upper one. Since they seem to be less contaminated, they should be managed through material recycling. The latter method is to submit the entire waste to the process of thermal transformation. The choice of pyrolysis as a method of thermal transformation of waste is not accidental, since more and more attention is being paid to its nature and ability to gain products in the form of gas, combustible oils, and charcoal. Although the Polish legislation requires the combustion of pyrolysis products, there are other ways to use these raw materials, such as for example in the production of methanol: R3 Recycling or the recovery of organic substances that are not used as solvents (including composting and other biological transformation processes). This heading also includes gasification and pyrolysis using these components as chemical reagents [2]. This article does not consider the possibility (due to the lack of literature data) of the return of waste floor panels to the manufacturers of these products and their possible inclusion in the production cycle under the so-called internal recirculation.

A methodology for determining the kinetic parameters of waste pyrolysis for fuels with an unidentifiable chemical formula of the basic molecule (e.g. post-consumer floor panel) has been proposed. The methodology can be helpful for comparative studies between various solid fuels and also waste with the general formula $C_xH_yO_z$. The location of the molecule, which was determined with this method, in the Van Krevelen diagram gives an excellent configuration of its energy status. This status can be changed subsequently through operations such as drying, mechanical drainage, heating, etc. in order to enhance energy properties (mainly calorific value by improving the ratio of fuel elements h/c (hydrogen/carbon) and o/c (oxygen/carbon)).

2. Thermodynamic and Kinetic Aspects of Pyrolysis

Pyrolysis is a process of thermochemical decomposition in an anaerobic atmosphere. It is a complex process of the decay of chemical compounds into smaller molecules under the influence of external thermal energy [12,13]. Most reactions that occur during pyrolysis are endothermic. In general, the pyrolysis process can be represented by reaction (1):



As a result of the pyrolysis, three fractions are formed [12,13]:

- the charcoal, consisting largely of carbon, also contains the mineral parts included in the substrate pyrolyzed;
- liquid fraction, consisting of an organic part with a complex chemical composition and water part containing, among others, acetic acid or methanol;

- gas fraction, which is a mixture of CO₂, CO, H₂, and hydrocarbons, mainly methane.

The literature presents different conditions for the process and the impact of these parameters on the products obtained [14–16]. The basic division into types of pyrolysis is related to the rate of heating of the fuel molecule. Slow pyrolysis occurs when the time required to warm the molecule to the pyrolysis temperature is significantly longer than the characteristic pyrolysis reaction time (heating \gg τ). When the situation is reversed, this process can be defined as fast pyrolysis. Other parameters that affect the quality and quantity of products are: a maximum temperature, the residence time of the primary decomposition products in the conversion zone, a degree of fuel fragmentation, the reactor heating method, and its construction. Table 1 presents types of pyrolysis and characteristic parameters influencing the decomposition, while Figure 1 shows the possibilities of the course of pyrolysis depending on the parameters in which the process is carried out.

Table 1. Types of pyrolysis and characteristic parameters influencing the decomposition [13].

Type of Pyrolysis	Residence time of Primary Decomposition Products in the Conversion Zone	Heating Rate [K/s]	Final Temperature [K]	Products
Carbonization	days	very slow	400	charcoal
Conventional	5–30 min	slow	600	charcoal, gas, liquid fraction
Fast	<2 s	very fast	500	liquid fraction
Very fast	<1 s	very fast	1000	reagents, gas
Underpressure	2–30 s	fast	400	liquid fraction

Pyrolysis as a method of obtaining fuels is a promising technology for the conversion of indirect wood-based waste such as floor panels. This type of waste can be found in the waste catalogue under the codes: 03 01 05, 17 09 04, and 20 01 38. The code allocation depends on the source of the waste floor panels [17].

Pyrolysis is a very complex process and, depending on the parameters of its operation, it is possible to obtain solid, liquid, and gas products in various weight ratios. The schematic diagram of the possibilities of the process, depending on the pressure, temperature, and speed of the process, was presented in work [13,18].

2.1. Heat Demand for the Pyrolysis Process

Although basically pyrolysis is an endothermic process, during its duration, there are effects of both heat extraction and its emission. At the time of pyrolysis, pyrogenetic water is formed, which is the result of the reaction of hydrogen with oxygen, as well as the result of the disintegration of hydroxyl group side chains. However, decomposition reactions are not the only chemical processes that occur during pyrolysis. The gases formed from the solid penetrate the remaining part of the solid phase (most often the char) and react with them. Therefore, it is difficult to determine clearly what is the thermal effect of the pyrolysis itself, and what are the effects of secondary reactions occurring between pyrolytic gases and the surface of the char [13]. The energy balance of the pyrolysis process is prepared in accordance with the first law of thermodynamics, from which it follows that if any transformation in the system changes its state and the system exchanges energy with the environment only through heat transport, then the relationship follows the equation below (2) [19]:

$$Q = \Delta h \quad (2)$$

where:

Δh – increase of enthalpy of the system

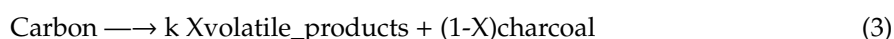
Q – heat exchanged with the environment

The above indicates that the change of enthalpy of the system is equal to the heat supplied, the quantity of which corresponds simultaneously to the heat demand for pyrolysis. In this case, possible heat losses to the environment are not taken into account. Thus, it is possible to use the calculation methodology proposed by [19], which is based on the balance of the enthalpy of substance formation, and takes into account the physical enthalpy of products in appropriate physical states.

Pyrolysis heat is determined on the basis of temperature measurements in DSC (Differential scanning calorimetry) scanning calorimeters. The results obtained in this type of device are a net measure of pyrolysis and secondary reactions. Pyrolysis heat is the difference in the energy of the formation of reagents and products at the reference temperature (298.15 K). The heat values of pyrolysis reported in the bibliography differ significantly from 750 kJ/kg (endotherm) to 130 kJ/kg (exothermic reaction). For example, pyrolysis heat in the range from 274 kJ/kg to 353 kJ/kg was determined using DSC [13]. Bibliography [13] presents thermogravimetric measurements for pine wood up to 700 °C, with a heating rate of 20 °C/min. After cooling, the samples of charcoal being still in the thermogravimeter were heated once again at the same rate. Shown the power consumed by pine wood and the charcoal during pyrolysis in TGA (Thermogravimetric analysis) as a function of temperature. After integration in time with the power difference consumed by the material, the thermal effect of the process is obtained. The difference among the thermal effects for wood and charcoal relates to pyrolysis heat, and it amounts to 1473 kJ/kg. This effect is clearly higher than the effect resulting from the above data.

2.2. Kinetics of the Pyrolysis Process

Fuels formed on the basis of waste floor panels are a complex solid substance, and there are thermal transformations in the hard-to-define structure of C–H–O–N molecules (which changes texture during the process) because it is difficult to implement a model description including reaction range, autocatalytic effects, diffusion effects, and structural changes. In the bibliography, one can find various approaches to pyrolysis kinetics; however, the basis for any analysis is the distribution reaction model, which occurs according to the following entry (3). This is as a single irreversible reaction of the thermal decomposition of carbon, but it is also helpful in case of a carbon-rich substance:



This entry is often regarded in the literature of the subject as too simplified, because it omits important transitional stages of the substance distribution, including, first of all, subsequent and possibly parallel reactions of the distribution of primary products, such as coal tar (metaplast). For this reason, many forms of kinetic equations have been formulated, also by dividing pyrolysis into individual stages. However, a comparison of various schemes of the decomposition of organic carbon leads to the conclusion that regardless of the method of receiving final products, the formula expressed by the dependence (3) reflects the final result of pyrolysis. As a consequence, many authors approximate the process of decomposition of a substance rich in organic carbon with the first-order reaction occurring uniformly in the entire volume of the particle [20]. The speed of extracting volatile parts can be described in this case by the Formula (4):

$$dV/dt = k (V_0^{daf} - V) \quad (4)$$

The most common source of information on pyrolysis kinetics are data obtained from thermogravimetric and derivative thermogravimetric measurements (TG/DTG). The results of such measurements under dynamic conditions indicate the existence of several process stages characterized by different quantities of apparent activation energy. The stages of various kinetics are attributed to the pyrolysis of the individual components of which the floor panels are made, including: wood-based HDF (high-density fiberboard) boards, urea–formaldehyde resins, and laminates [21].

2.3. Van KREVELEN Systematics and Wood-Based Waste

The original intention and achievement of Van Krevelen [22] was the presentation of classical fuels in a plane coordinate system, where the atomic quotients of O/C were given on the abscissa x and on the ordinates $y = H/C$ (excluding N, S, P, and micronutrients). Assuming that carbon is tetravalent, the maximum value of the ordinate is 4, and the abscissa $x = O/C \leq 2$. The Van Krevelen diagram was later modified by Meunieur [23], who adopted the total chemical formula CH_yO_x (x, y coordinates from the diagram) for these fuels. Modification made by Meunieur showed the chemical structure more accurately than the percentage mass fraction of elements C, H, and O [24].

When analyzing wood-based waste (and knowing their chemical composition), it was possible to determine their picture in the Van Krevelen systematics. The aim was to define the place of this waste and its energy suitability in comparison to classical fuels. To determine the waste position in the Van Krevelen diagram, it is necessary to calculate the atomic quotients x and y for dry organic matter of waste without sulfur and phosphorus in accordance with the condition:

$$C + N + O + H = 100\% \quad (5)$$

where:

C, N, O, and H – content of elements in the sample in percentage by weight.

At the stage of laboratory tests, it was possible to determine the content of all four elements required by the procedure. To find an illustration/point in the diagram for waste, it is necessary to separate the content of these elements [24,25]. Determination of the empirical formula of wood-based waste ground on the percentage content of elements was based on the conversion of this content into molar of elemental atoms. The obtained values were divided by the lowest number in the ratio, which was in this case 1.071. Ultimately, the empirical formula is as follows:

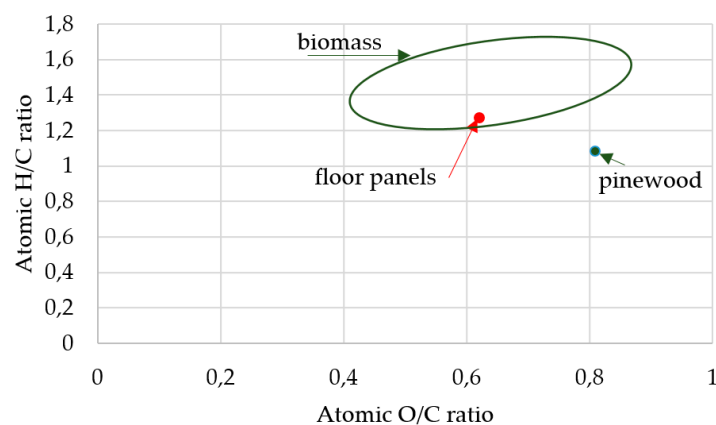


Figure 1. Illustration of the position of waste from floor panels in the Van Krevelen system (author's own study) [24,26].

Figure 1 presents the location of wood-based waste (floor panels) in the Van Krevelen diagram. The coordinates of the point characterizing wood-based waste were calculated according to the formulas [24,25]:

$$X = OM_c/CM_o = 0.62 \quad (7)$$

$$Y = HM_c/CMH = 1.27 \quad (8)$$

where:

MC, MH, and MO – molar masses of carbon, hydrogen, and oxygen;

C, H, O – content of elements in the dry organic mass of the residue in percentage by weight.

3. Materials and Methods

Tests TG-MS/FTIR were carried out on samples with the following assay [27]:

- ZMT/20/2019 (pinewood)
- ZMT/21/2019 (bottom layer of the floor panel)
- ZMT/22/2019 (middle layer made of HDF)
- ZMT/23/2019 (upper layer of the floor panel)
- ZMT/24/2019 (complete floor panel)
- ZMT/25/2019 (urea–formaldehyde resin)

These tests were carried out by means of the STA 409 PG Luxx thermogravimetric analyzer from Netzsch on a TG (Thermogravimetry) carrier coupled with a quadrupole mass spectrometer QMS Aeolos and a medium infrared spectrometer FTIR (Fourier Transform Infrared Spectroscopy) Tensor 27 from Bruker. The tests were carried out in an argon atmosphere (purity class 5.0, flow 25 mL/min), from 40 to 1000 °C, with a heating rate of 10 K/min. The sample weight was 10 ± 0.1 mg. The test was performed in crucibles with Al₂O₃ with a lid. In order to eliminate traces of oxygen in the thermal mixer, the OTS (Oxygen Trap System) system was used. The measurement was corrected with corrective measurements (measurement without a sample). The corrective measurements were carried out three times.

This paper presents an analysis of the physicochemical properties of selected post-consumer wood waste and urea–formaldehyde resin. The goal of the analysis was to determine specific fuel properties, such as moisture, ash content, content of volatile components, calorific value, and elemental composition (C, H, O, N, S, Cl).

The characteristics of the physicochemical properties of the tested samples are presented in Table 2.

Table 2. Physicochemical properties of samples.

Parameter	ZMT/20/2019	ZMT/21/2019	ZMT/22/2019	ZMT/23/2019	ZMT/24/2019
Total humidity, % mass	13.81	5.35	6.07	5.20	6.16
Combustible fraction, % mass	98.36	99.39	99.75	95.88	99.12
Ash, % mass	1.64	0.61	0.25	4.12	0.88
Volatile fraction, % mass	78.17	80.28	82.09	77.43	82.07
Heat of combustion, MJ/kg	20.23	19.63	18.70	18.88	19.89
Calorific value, MJ/kg	18.93	17.96	17.10	17.46	18.58
Carbon, % mass	44.94	53.23	51.72	46.51	48.34
Hydrogen, % mass	4.25	6.85	6.44	5.71	5.11
Oxygen, % mass	48.56	33.09	35.85	35.20	40.24
Nitrogen, % mass	0.61	5.99	5.46	8.08	5.19
Sulfur, % mass	0.003	0.23	0.28	0.38	0.24
Chloride, % mass	ppo	ppo	ppo	ppo	ppo

Ppo—below the limit of quantification.

The individual layers of the floor panel are characterized by similar humidity of 5–6%, while pine wood is characterized by humidity at the level of approximately 14%. All the samples had a low ash content of less than 4.12% and a high content of volatile matter in the range of 78–82%. The wastes are characterized by a calorific value at a similar level, i.e., above 17 MJ/kg. In all the layers of the panel, there was a high nitrogen content in the range from 5.19–8.08%, while a low nitrogen content was recorded in pine wood. The tested wastes had low sulfur and chlorine content.

4. Results and Discussion

Figure 2 shows the weight loss curves for individual samples. It reveals the unexpected similarity between the weight loss curves of the pure wood sample and the sample representing the upper layer of the panel, as well as three curves of samples: the bottom and the middle layers and the sample of the averaged complete panel composition. In the case of waste floor panel pyrolysis, usually three stages of thermal decomposition are identified: in the first stage (from environment temperature to about 140–180 °C), the samples lose moisture; in the second stage (from about 160 to 400 °C), the material pyrolysis takes place; the third stage, which is clearly slower than the preceding one, occurs at a temperature of about 400 to 700 °C. The divergence from the three-stage decomposition was observed at the sample of a urea–formaldehyde resin, which decomposed in five stages occurring in the following temperature ranges: 40–120 °C, 120–200 °C, 200–260 °C, 260–300 °C, and 300–450 °C.

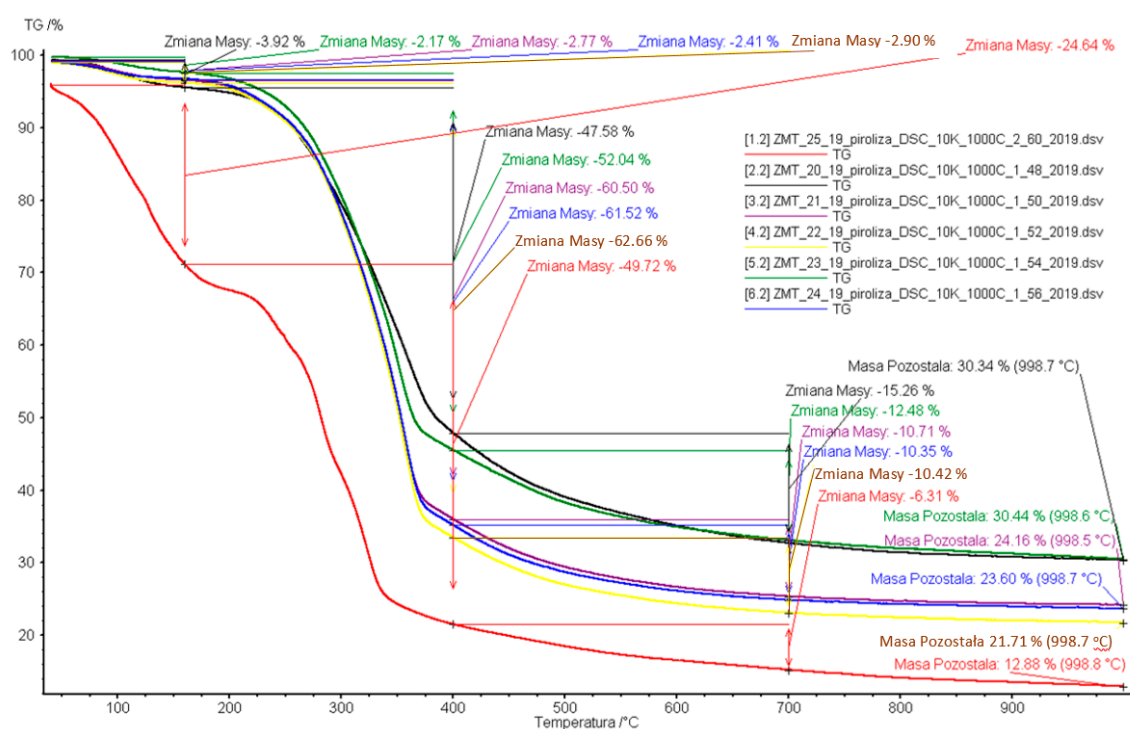


Figure 2. TG (thermogravimetry) curves of all the samples tested (own research).

Figure 3 shows the weight loss rates of individual samples. All the samples of different layers of the floor panel have a similar velocity of mass loss ranging from 6.145 to 7.792%/min, and the largest distribution occurs at the temperatures of 35.4 to 357.1 °C. This creates good conditions for simultaneous decomposition at almost the same temperature. A slightly different rate of decay characterizes clean wood and it amounts to 4.097%/min. The chemical additives in the panel increase the weight loss rate almost twice.

Figures 4–6 present representative results regarding TG, DTG curves, and ionic current (MS). Studies show that water (ion $m/e = 18$, blue line) is emitted from ZMT/20–24/2019 samples in two stages: when the samples lose moisture in the temperature range from the environment temperature to about 180 °C, and in the range from about 250 to 450 °C. The other main products of pyrolysis, such as: carbon monoxide ($m/e = 28$, purple line), carbon dioxide ($m/e = 44$, yellow line), methane ($m/e = 16$, red line), and nitric oxide ($m/e = 30$, green line) are emitted from samples in the range from about 200 to 500 °C. In the case of the ZMT/25/2019 sample (urea–formaldehyde resin), the temperature ranges in which individual products are released are slightly different, and also, ions were recorded: $m/e = 17$ (most likely ammonia – light green line) and $m/e = 57$ (light blue line).

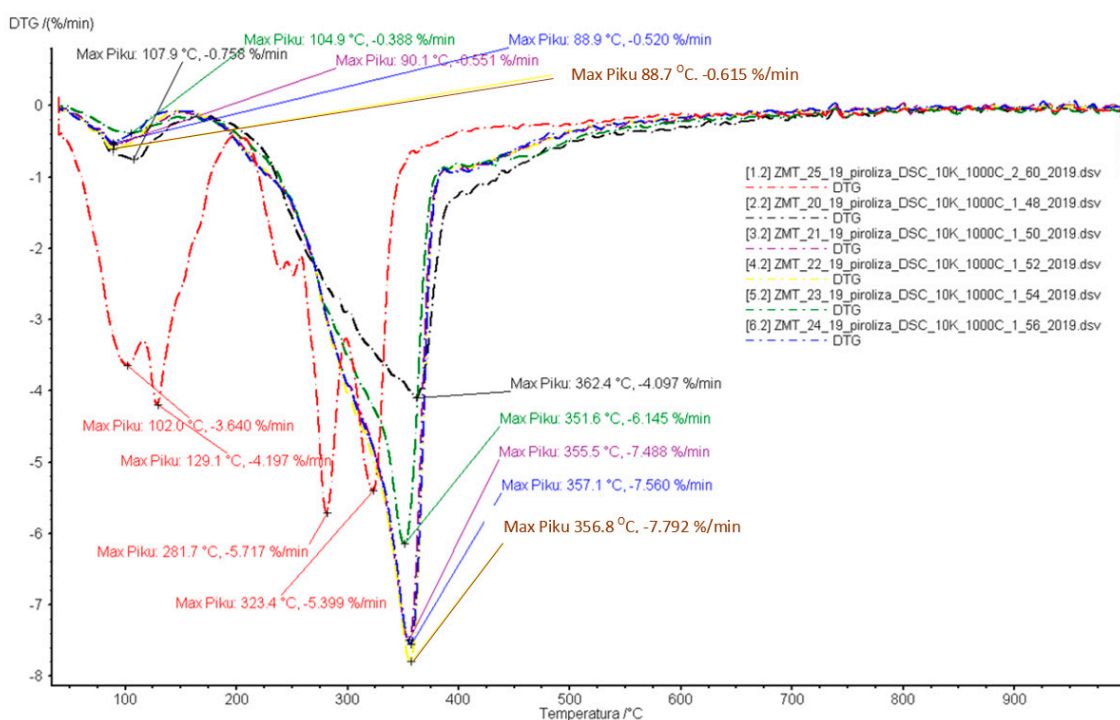


Figure 3. Derivative thermogravimetric measurements (DTG) curves of all samples tested (own research).

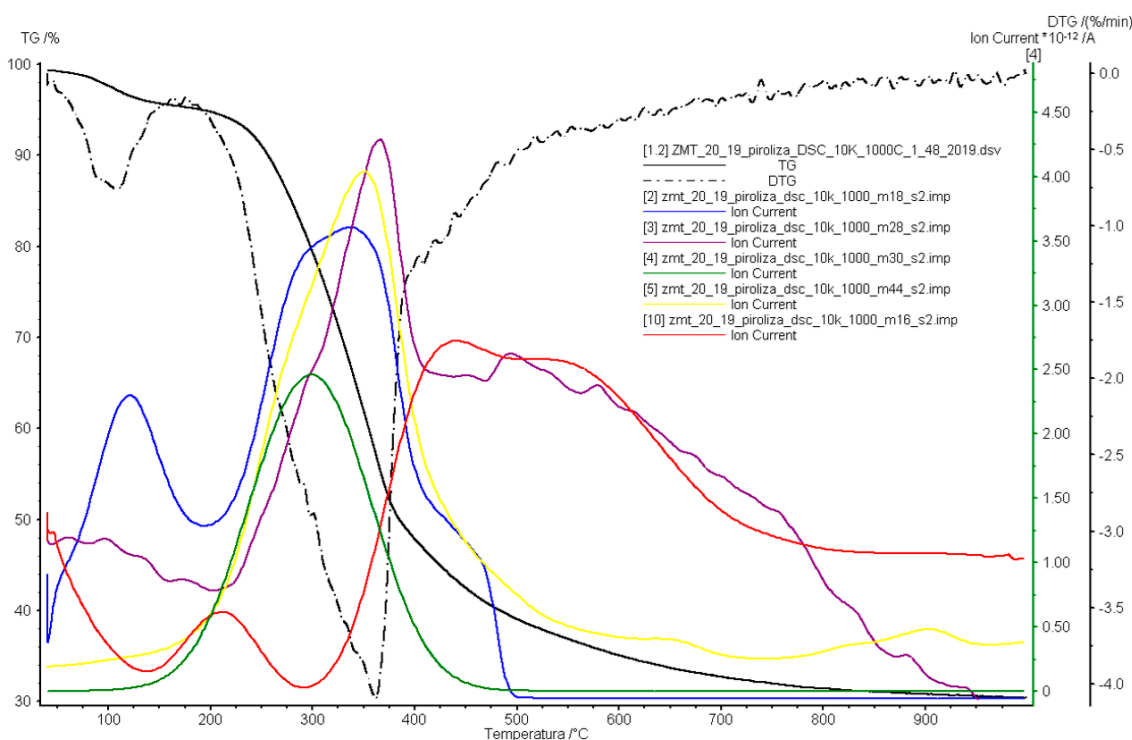


Figure 4. TG and DTG, curves and ion current intensity for the sample ZMT/20/2019 (own research).

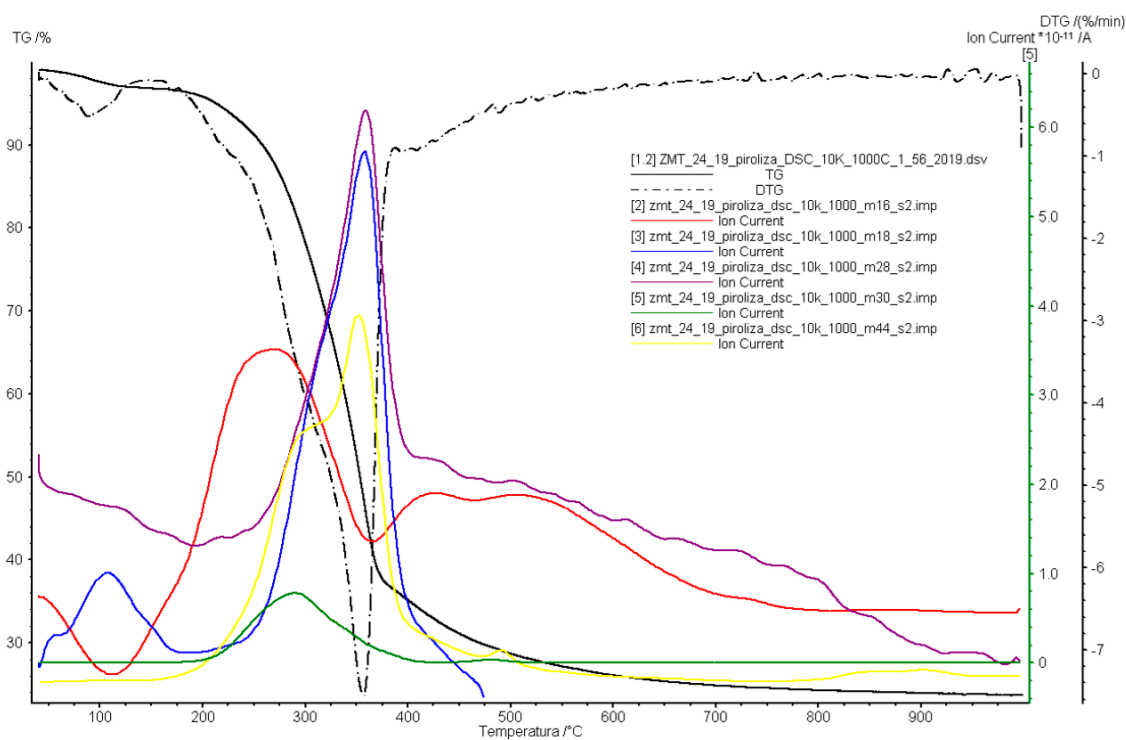


Figure 5. TG and DTG curves, and ion current intensity for the sample ZMT/24/2019 (own research).

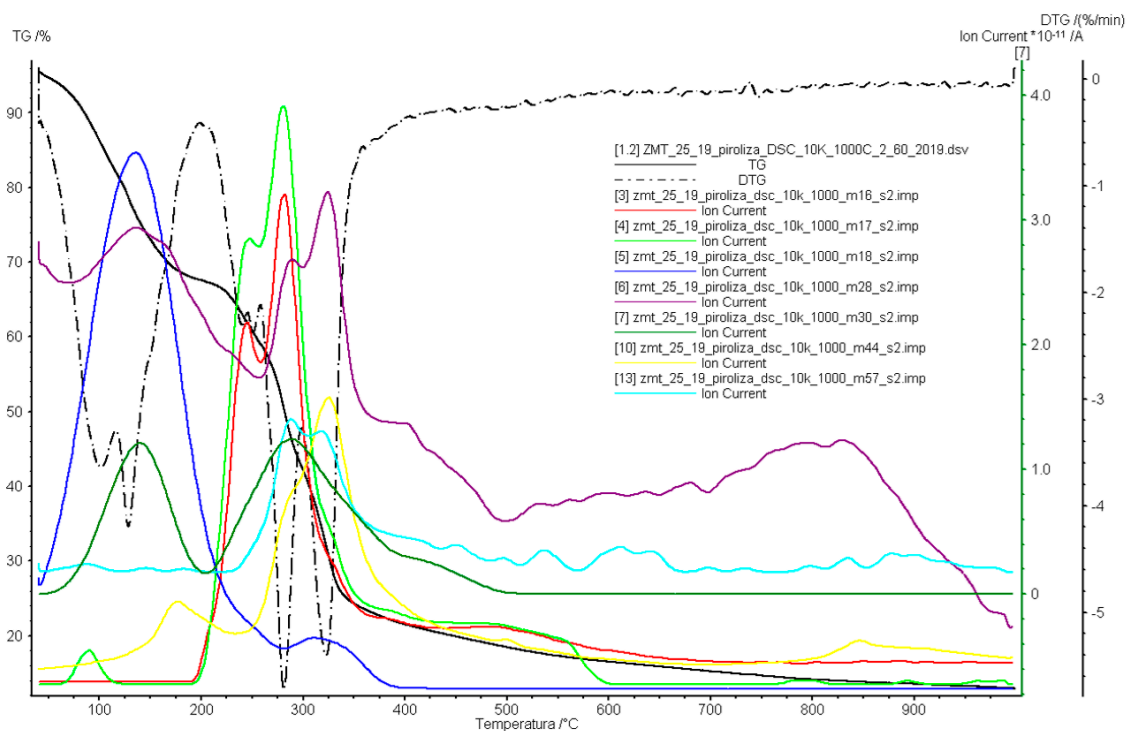


Figure 6. TG and DTG curves, and ion current intensity for the sample ZMT/25/2019 (own research).

The combination of thermoanalysis with the analysis of gaseous products has provided knowledge of the mechanisms that occur in the examined waste during thermal processes and will enable the qualitative assessment of gases emitted during heating with regard to the chemical composition.

Figure 7a,b show the course of the curves of the emission of selected gases as a function of temperature with the TG curves for the sample of the charcoal by using the FTIR spectrometer data

during the pyrolysis process of the samples: ZMT/20/2019 and ZMT/24/2019. It is clearly evident that ammonia and nitrogen dioxide are emitted during the pyrolysis process of the waste floor panel. This phenomenon is not observed in the case of the pyrolysis of pure pine wood.

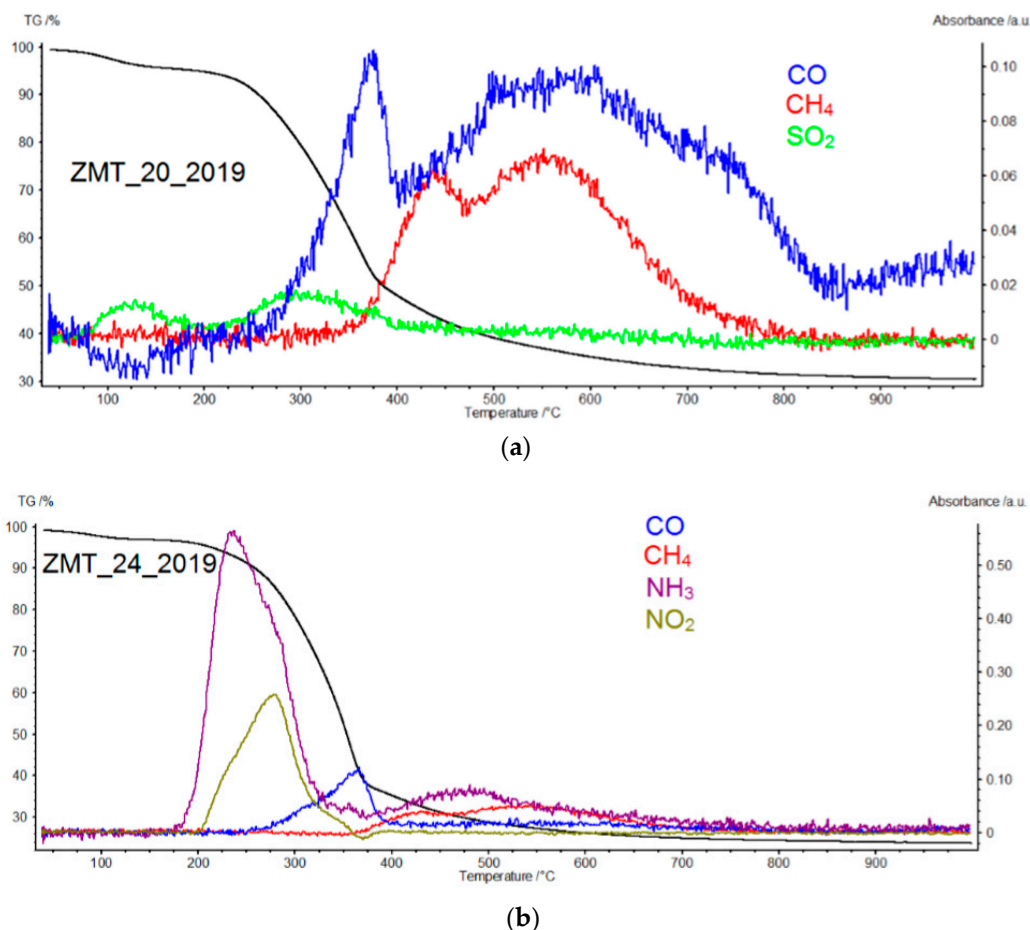


Figure 7. TG curve and curves of emissions of carbon monoxide, methane, nitrogen dioxide, and ammonia for the samples (a) ZMT/20/2019 and (b) ZMT/24/2019 (own research).

This phenomenon is caused by the addition of urea–formaldehyde resin to the floor panels, which contains about 37% nitrogen in the composition [28].

The products of pyrolysis depending on the conditions of the process (which include heating speed, temperature, residence time, granulation of the material, etc.) are as follows: gas (CO, CO₂, CH₄,...), pyrolysis oil, and carbonizate (carbonized solid). For example, increasing the heating rate directs pyrolysis products toward the production of bio-oil, and long-term heating is used to produce carbonized solids. All pyrolysis products have a positive chemical enthalpy (calorific value), so they can be used for energy, e.g., in the production of heat and electricity or cogeneration. However, pursuant to the Polish legislation, products of the waste pyrolysis process should ultimately be oxidized and incinerated. There is also a possible legal path for another use of pyrolysis gases for the production of chemicals, e.g., in methanol production.

5. Conclusions

Mechanical separation of the panel layers for the purpose of different management of the upper layer (thermal transformation) and lower layers (recycling) is not reasonable. This thesis is justified by the following:

- (a) All samples, regardless of the location of the layer (upper/middle/lower), emit, according to the TG curve, high amounts of the NO_x and NH_3 , which are derivatives of the decomposition of urea–formaldehyde resin, as shown in Figures 4–7;
- (b) The sample of pure pine wood does not generate NO_x and NH_3 during thermal decomposition, as shown in Figures 4 and 7a;
- (c) The addition of the hardeners (glues) in the form of the urea–formaldehyde resin efficiently contaminates the entire cross-section of the panel.

The thermal transformation (combustion/gasification/pyrolysis) shall be indicated as the only reasonable method of management of floor panels. All these methods must deal with the issue of emissions of the contaminants as a result of decomposition or the secondary reaction mechanisms generated by the addition of urea–formaldehyde resin.

The problem of the combustion of floor panels in the layer on the grate of the waste thermal treatment installation (ITPOK) was the subject of description in the publication [21,29–31].

The proposed methods of thermal decomposition of floor panels using pyrolysis indicate the advantages of this process as evidenced by:

- (a) Energy characteristics of the location of the formed fuel in the Van Krevelen diagram in the area of coal fuels and peat, as shown in Figure 1;
- (b) The DTG distribution curves, and peaks of the maximum mass loss rate of the panel samples and the temperature at which they take place, are similar in the area from 6.145 to 7.792%/min., in temperatures: from 351.4 to 357.1 °C, as shown in Figure 3;
- (c) The high content of volatile parts in Table 2 suggests a high tendency for thermal decomposition in accordance with Equation (4); a similar tendency is related to coals, which is documented in the publication [20]; and
- (d) A factor promoting the rate of decomposition of matter during pyrolysis is also a lower oxygen content in the panel, which is 40.24% by weight, compared to its content in pure wood, which is 48.56% by weight, as shown in Table 2. Lower oxygen content improves the energy qualities of the fuel.

The research on pyrolysis should be further developed with solutions reducing the NO_x and NH_3 emissions from this process.

Post-consumer wood in the form of floor panels, MDF boards, etc., is a hazardous waste due to the content of chemical substances; however, these substances improve their functional qualities (hardness, water resistance, etc.). In this study, an attempt was made to check how contaminated the waste is and whether at least part of it is suitable for such management that is different from thermal management. Analyses of subsequent sections of the tested waste showed almost equal contamination with urea–formaldehyde resin in the panel cross-section. Therefore, it is recommended to direct this waste to organized thermal disposal. Pyrolysis seems to be a good solution here, especially since its products, depending on the mode of its conduction and the heating rate applied, can be also used as raw material for the production of chemicals.

Kinetic studies should be related to the thermodynamic analysis of pyrolysis. It is suggested to continue research on the basis of compounds: the enthalpy of creating substrates—which is an energy of process activation—oxygen content, and volatile parts in substrates, as well as the heating rate in the process (type of reactor for conducting pyrolysis).

Author Contributions: Conceptualization, T.J.; formal analysis, T.J.; writing, T.J.; funding acquisition for research, M.K.-S.; investigation M.K.-S.; project administration M.K.-S.; resources M.K.-S.

Funding: This research was funded by the InnoEnergy. Its publication was supported as part of statutory research carried out at the Silesian University of Technology 08/030/BK_18/0045.

Conflicts of Interest: The authors declare no conflict of interest.

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