

MICROSTRUCTURAL ANALYSIS AND IMAGE-BASED EVALUATION OF TEMPERATURE-INDUCED TRANSFORMATIONS IN BIOMASS DURING TORREFACTION

Serhii Yermakov^{1,2}, Julia Galchynska¹, Pavlo Fedirko¹, Zoya Pustova¹, Oleksii Sikora¹

¹Podillia State University, Ukraine; ²Kamyanets-Podilskyi Instrument Applied College of Construction, Architecture and Design, Ukraine
ermkov@gmail.com, dakgps@gmail.com

Abstract. This paper presents the results of a microstructural analysis of temperature-induced transformations in biomass during torrefaction. The study is aimed at identifying the regularities of internal and surface structural changes in lignocellulosic raw materials depending on thermal treatment regimes, as well as at assessing the feasibility of using image-based methods for evaluating the degree of torrefaction. The investigated material included woody biomass (poplar), which was selected as a representative lignocellulosic feedstock. Microstructural observations revealed that increasing torrefaction temperature leads to successive and well-defined structural transformations manifested by gradual degradation of cellular and fibrous architecture, formation of cracks and pores, and transition to a predominantly carbonized matrix. It was established that these transformations occur progressively with temperature increase, reflecting the stages of thermal decomposition of biomass components. The obtained results indicate that the degree of torrefaction can be qualitatively and quantitatively assessed based on structurally observable features. In particular, image-based analysis of microstructural changes allows for the evaluation of structural degradation through parameters such as edge density. The quantitative analysis showed that the edge density increased from 15.62% in raw biomass to 34.33% at 260 °C, indicating intensive structural degradation. At the same time, mass loss increased significantly at temperatures above 220–240 °C, corresponding to the stage of active thermal decomposition. The proposed approach demonstrates that image analysis can be considered a simple and informative tool for evaluating torrefaction efficiency and the depth of thermal transformation of biomass.

Keywords: torrefaction, biomass, microstructure, bioenergy, energy efficiency.

Introduction

The growing interest in renewable energy sources necessitates improving the efficiency of biomass utilization as a fuel. One of the most promising methods of thermochemical upgrading is torrefaction, a mild thermal treatment process carried out in an inert environment that enhances fuel properties, reduces moisture content, and increases the energy density of biomass [1; 2].

The assessment of the degree of torrefaction and optimization of thermal treatment parameters have been widely investigated, particularly for various types of agricultural biomass. Previous studies have demonstrated that temperature and residence time significantly influence mass loss and the energy characteristics of the resulting solid product [3-5].

During torrefaction, partial degradation of hemicellulose and cellulose occurs, leading to changes in chemical composition and physical properties of biomass [6; 7]. As a result, the material becomes more brittle and exhibits improved grindability, which is important for further utilization in combustion and gasification processes [8].

In addition to chemical transformations, microstructural changes play a crucial role. It has been shown that torrefaction leads to the destruction of cell walls, formation of microcracks, and an increase in structural heterogeneity of biomass [9; 10]. These changes directly affect the reactivity of the material, as they determine the availability of active surface area and influence heat and mass transfer processes [11].

Conventional approaches to structural characterization include microscopy and porosity measurements; however, their application may be limited, especially for carbonized materials with reduced image contrast [12]. Therefore, increasing attention is being paid to digital image processing techniques, particularly texture analysis methods [13].

Edge detection techniques are widely used to identify structural features such as cracks and boundaries formed during thermal degradation. These methods enable the transition from qualitative observation to quantitative evaluation of structural complexity [14].

Despite the considerable number of studies on biomass torrefaction, the quantitative assessment of microstructural transformations and their relationship with thermal degradation remains insufficiently

explored [15]. In particular, limited attention has been given to the use of image-based parameters as indicators of biomass reactivity.

Furthermore, in the context of modern energy strategies and development of the bioeconomy, improving the efficiency of biomass utilization becomes increasingly important [16-18].

The aim of this study is to investigate the effect of torrefaction temperature on microstructural transformations of poplar biomass and to develop an approach for their quantitative evaluation based on edge density as an image-derived parameter.

Materials and methods

Torrefaction procedure

Experimental studies were carried out in the educational and research laboratory “DAK GPS” of the higher education institution “Podilskyi State University”, which specializes in research in the field of biomass energy and methods of improving the energy properties of biomass [19-21].

Poplar wood was used as the raw material for the study, crushed using a hammer mill to a mulch-like state. The obtained material was characterized by a relatively uniform particle size distribution with a predominant particle size in the range of 5-15 mm and a small amount of fine fraction. Such a size ensured sufficient homogeneity of the samples for thermal treatment and subsequent microstructural analysis. Prior to the experiments, the samples were dried to an air-dry state to ensure stable initial conditions. The samples were prepared with the same size to minimize the influence of geometric factors on heat and mass transfer during torrefaction.

Torrefaction was carried out under laboratory conditions at temperatures of 180, 200, 220, 240, 260 and 300 °C. The residence time at the set temperature was 15 minutes. After completion of the thermal treatment, the samples were cooled to room temperature before further analysis. Mass measurements were performed using laboratory electronic scales with an accuracy of ± 0.01 g before and after torrefaction.

Mass loss was determined by the gravimetric method according to the equation:

$$ML = \frac{(m_0 - m_t)}{m_0} 100\% , \quad (1)$$

where m_0 – initial mass of the sample;
 m_t – mass after torrefaction.

Microstructural analysis and image processing

Structural changes in biomass were studied using optical microscopy. Micrographs were obtained for untreated and thermally treated samples under identical magnification and illumination conditions, ensuring the validity of comparison.

During the analysis, the following characteristics were evaluated: fiber integrity, presence of microcracks, pore development, and overall structural heterogeneity.

To enhance structural features, the micrographs were processed using an edge detection method based on the analysis of image brightness gradients.

The processing was performed using a standard edge highlighting filter, which allows the identification of areas with sharp intensity changes corresponding to the boundaries of structural elements, microcracks, and defects.

All images were processed using identical parameters to ensure reproducibility of the results. Edge density was calculated as the ratio of the number of pixels corresponding to detected edges to the total number of pixels in the image after binarization. Quantitative assessment of structural changes was carried out using the edge density parameter, defined as:

$$E = \frac{A_{edges}}{A_{total}} 100\% , \quad (2)$$

where A_{edges} – area of pixels corresponding to edges;
 A_{total} – total image area.

Before calculation, the images were converted to grayscale and binarized to separate edges from the background. The obtained values are relative indicators and are used for comparative analysis of structural changes.

The analysis of the results was carried out by comparing mass loss, microstructural changes, and the edge density parameter. This approach makes it possible to assess the effect of torrefaction temperature on the structure of biomass and to draw conclusions regarding its reactivity and technological properties.

Each experimental condition was repeated three times, and the reported values represent average results. The relative deviation of the measured parameters did not exceed 5%.

Results and discussion

Mass loss during torrefaction

Fig. 1 shows the dependence of mass loss of poplar biomass on torrefaction temperature at a residence time of 15 minutes. The mass loss values were calculated according to Eq. (1).

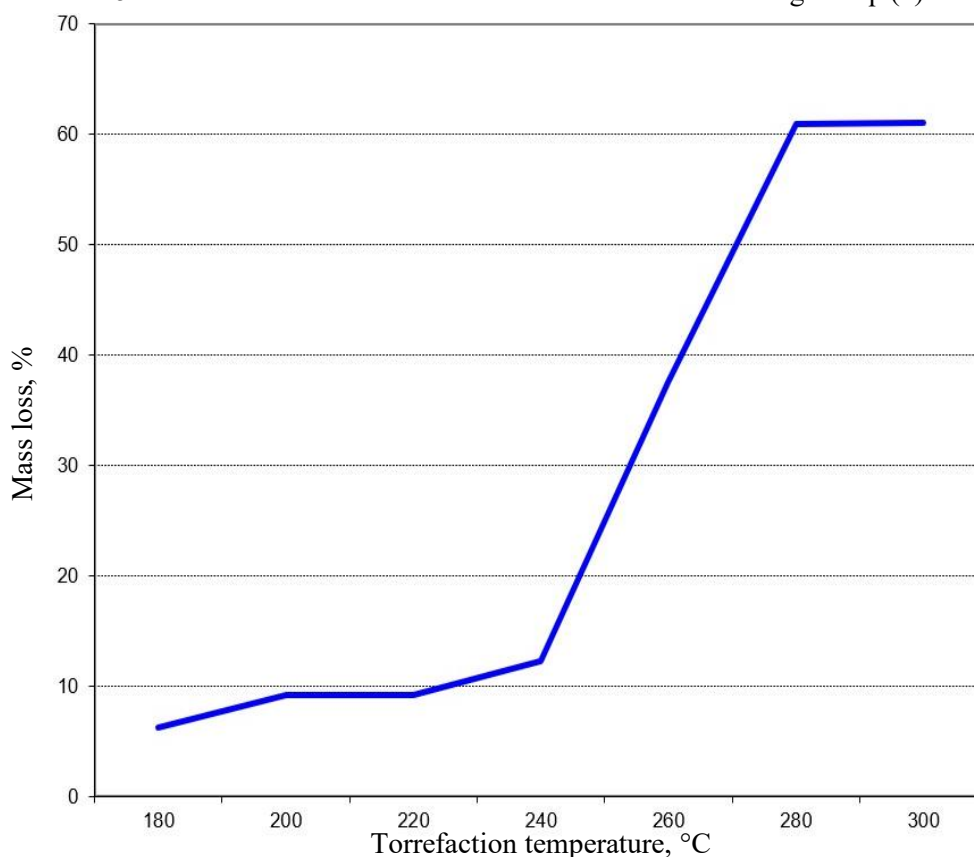


Fig. 1. **Dependence of mass loss of poplar biomass on torrefaction temperature** (residence time 15 min)

Mass loss increased with increasing the torrefaction temperature, indicating gradual thermal decomposition of the main biomass components. In the temperature range up to 220 °C, the changes are relatively small and are mainly associated with hemicellulose degradation. With further temperature increase (above 220-240 °C), a significant rise in mass loss is observed, which is caused by active release of volatile components and partial decomposition of cellulose.

The visual appearance of poplar biomass samples before and after torrefaction is shown in Fig. 2.

With increasing the torrefaction temperature, a gradual change in sample color from light brown to dark brown and almost black is observed. This is associated with an increasing degree of carbonization and removal of volatile organic compounds.



Fig. 2. Visual appearance of poplar biomass before and after torrefaction at different temperatures

Analysis of microstructural changes based on optical microscopy and edge detection

Micrographs of poplar biomass at different torrefaction temperatures are shown in Fig. 3a. Microscopic analysis shows significant structural changes in biomass during torrefaction. The raw sample is characterized by a relatively intact and homogeneous structure. With increasing temperature, gradual destruction of the cellular structure, fiber delamination, and formation of microcracks are observed.

In the temperature range of 200-220 °C, initial structural degradation occurs, whereas at temperatures above 240 °C the structure becomes significantly more fragmented and heterogeneous, indicating deep thermal decomposition.

To better identify structural changes, an edge detection method was applied. The corresponding images are shown in Fig. 3b.

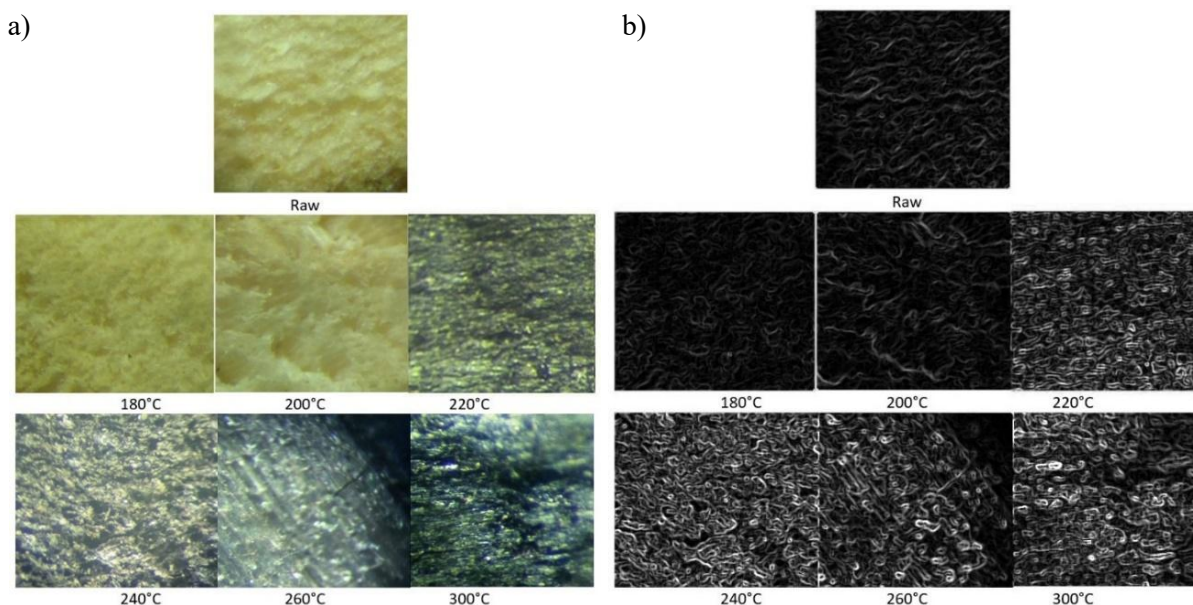


Fig. 3. Microstructure of poplar biomass at different torrefaction temperatures:
a – original micrographs; b – processed images (edge detection)

Image processing makes it possible to visualize the development of microcracks, boundaries of structural elements, and heterogeneities that are not always clearly visible in the original micrographs.

With increasing the temperature, a significant increase in the intensity and number of detected edges is observed, indicating a higher degree of structural degradation. The most pronounced changes are

observed at temperatures of 240-300 °C. The results of the quantitative evaluation of the poplar biomass structure, determined using the edge density parameter calculated according to Eq. (2), are presented in Table 1.

Table 1

Comprehensive assessment of structural changes in poplar biomass during torrefaction

Temperature, °C	Structure characteristics	Edge density, %
Raw	Intact, homogeneous structure	15.62
180	Minor deformations, isolated cracks	15.82
200	Appearance of microcracks, initial delamination	13.11
220	Developed crack structure	15.90
240	Fragmentation, high heterogeneity	21.60
260	Strong structural degradation	34.33
300	Carbonized, porous structure	32.89

It should be noted that the edge density parameter does not increase monotonically with increasing temperature but exhibits slight fluctuations. This behavior may be caused by several factors, including the natural heterogeneity of biomass structure, transitional stages of thermal degradation, and limitations of image analysis methods. In the intermediate temperature range, partial structural rearrangement of the material is possible, leading to temporary changes in the intensity of detected edges. In addition, at high temperatures, an increased degree of carbonization may reduce the image contrast, affecting the efficiency of edge detection. Thus, the edge density parameter should be considered as a relative indicator of structural heterogeneity rather than an absolute measure of porosity or the degree of material degradation.

The mass loss values represent the gravimetric changes occurring during torrefaction, whereas the image-based analysis reflects microstructural transformations of the biomass. These two approaches characterize different aspects of the process: the gravimetric analysis quantifies the removal of volatile components, while the image analysis reveals the structural degradation associated with these transformations. The comparison of these parameters allows indirect evaluation of the relationship between chemical degradation and microstructural transformation.

Quantitative analysis of the edge density parameter calculated according to Eq. (2) shows that its value remains relatively stable at temperatures up to 220 °C, corresponding to minor structural changes, whereas starting from 240 °C a sharp increase in this parameter is observed, indicating intensive formation of microcracks and structural degradation of biomass. Maximum values are reached at temperatures of 260-300 °C, corresponding to the stage of deep thermal decomposition and formation of a highly heterogeneous carbonized matrix. A slight decrease at 300 °C may be associated with the formation of a more uniform carbonized structure and reduced image contrast.

The obtained results demonstrate a clear relationship between torrefaction temperature, mass loss, and microstructural changes in biomass. An increase in mass loss is accompanied by the development of microcracks, increased structural heterogeneity, and material fragmentation, which in turn leads to an increase in the available reactive surface. Thus, the edge density parameter can be considered as an indirect indicator of biomass reactivity, as well as its grindability and suitability for further thermochemical conversion.

The microstructural changes identified in this study are consistent with results reported in the literature, where torrefaction leads to destruction of the cellular structure of wood, development of microcracks, and increased brittleness of the material [8; 9]. The applied image analysis approach based on edge detection makes it possible to quantitatively evaluate the degree of structural heterogeneity, and similar texture analysis methods are widely used for characterization of complex materials [13]. The non-monotonic behavior of edge density is also consistent with other studies of digital microstructure analysis, where the influence of material heterogeneity and image formation conditions on quantitative indicators has been noted [22]. Thus, the obtained results confirm that edge density analysis can be used as an indicator of structural degradation of biomass during torrefaction.

Conclusions

1. The effect of torrefaction temperature on the microstructural transformations of poplar biomass was investigated using image-based analysis methods.
2. It was established that increasing the torrefaction temperature leads to progressive structural degradation of biomass, manifested by fiber delamination, formation of microcracks, and increased structural heterogeneity. The most intensive structural changes occur at temperatures above 240 °C, where the edge density increases from 15.90% (220 °C) to 21.60-34.33% (240-260 °C).
3. The application of edge detection methods allows effective identification and quantitative evaluation of structural changes in biomass. The proposed edge density parameter is sensitive to structural transformations and can be used as an indicator of the degree of thermal degradation.
4. The edge density parameter generally increases with increasing the torrefaction temperature, although it exhibits non-monotonic behaviour due to material heterogeneity, transitional degradation stages, and limitations of image processing.
5. A clear relationship between mass loss and microstructural changes was established, associated with increased fragmentation and development of reactive surface area, which may enhance biomass reactivity and grindability.
6. The proposed image-based approach can be used to evaluate the efficiency of biomass thermal treatment and to optimize technological parameters of its processing.

Author contributions

Conceptualization, S.Y. and Y.G.; methodology, S.Y. and P.F.; software, S.Y., Z.P. and P.F.; validation, Y.G. and O.S.; formal analysis, Y.G., S.Y. and P.F.; investigation, S.Y., P.F., Z.P. and O.S.; data curation, S.Y., Y.G.; writing – original draft preparation, S.Y., Z.P. and O.S.; writing – review and editing, S.Y. and Y.G.; visualization, P.F., Z.P., O.S.; project administration, S.Y.; funding acquisition, O.S.

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