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THE EFFECT OF TORREFACTION ON PELLETS MADE FROM VEGETAL BIOMASS GENERATED BY FRUIT SHRUBS

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Abstract. This study investigates the effects of torrefaction on the properties of pellets produced from vegetable biomass residues of fruit shrubs. The research focuses on two pellet formulations: (1) a blend of sea buckthorn (*Hippophae rhamnoides* L.) and blackberry (*Rubus fruticosus*) residues and (2) a mixture of sea buckthorn residues with wheat straw. The torrefaction process was conducted in an inert argon atmosphere at temperatures between 200 °C and 300 °C for durations ranging from 10 to 30 min. The study assessed the impact of these parameters on the net calorific value and ash content of the pellets. Results indicate that torrefaction significantly enhances the calorific value of the biomass, with optimal conditions identified at 250-280 °C and exposure times of 15-25 min. However, excessive temperature and duration lead to mass losses and increased ash content. These findings provide insights into optimizing torrefaction parameters for improving the quality of densified solid biofuels derived from agricultural residues.

Keywords: *torrefaction, fruit shrub biomass, solid biofuels, calorific value, ash content.*

Rezumat. Acest studiu investighează efectele torefierii asupra proprietăților peleților produși din reziduuri de biomasă vegetală provenite de la arbuști fructiferi. Cercetarea se concentrează pe două formulări de peleți: (1) un amestec de reziduuri de cătină albă (*Hippophae rhamnoides* L.) și mur (*Rubus fruticosus*) și (2) un amestec de reziduuri de cătină albă cu paie de grâu. Procesul de torefiere a fost realizat într-o atmosferă inertă de argon, la temperaturi cuprinse între 200 °C și 300 °C, pentru durate de expunere între 10 și 30 min. Studiul a evaluat impactul acestor parametri asupra valorii calorifice nete și conținutului de cenușă al peleților. Rezultatele indică faptul că torefierea îmbunătățește semnificativ valoarea calorifică a biomasei, condițiile optime fiind identificate la temperaturi de 250 - 280 °C și durate de expunere de 15 - 25 min. Totuși, temperaturile și timpii excesivi conduc la pierderi de masă și la creșterea conținutului de cenușă. Aceste constatări oferă informații utile pentru optimizarea parametrilor de torefiere în vederea îmbunătățirii calității biocombustibililor solizi densificați obținuți din reziduuri agricole.

Cuvinte cheie: *torefiere, biomasă de arbuști fructiferi, biocombustibili solizi, valoare calorifică, conținut de cenușă.*

1. Introduction

The cultivation of fruit shrubs is an essential sector of agriculture, contributing to the diversification of agricultural production and generating additional income for farmers [1]. In the Republic of Moldova, due to its geographical position and morphological characteristics, especially in the Northern and Central regions, the areas designated for these crops have seen significant growth, increasing from 698 ha in 2021 to 908 ha in 2023 [2].

The results obtained in our previous research demonstrate that fruit shrubs generate a significant amount of plant biomass, which can be successfully used for the production of densified solid biofuels, including pellets [3-6]. The use of these residues as raw material for pellet production holds significant potential for the development of renewable energy in the Republic of Moldova.

Wood pellets are successfully used for heating in both the residential and industrial sectors [7]. However, the production of pellets from agricultural residues, including plant biomass from fruit shrubs, has some disadvantages, such as hydrophilicity, low calorific value, and poor densification capacity [8,9].

Thermochemical treatment through torrefaction can eliminate or at least reduce these disadvantages by significantly modifying certain essential characteristics [10]. Torrefaction is a thermal treatment process in which biomass, before densification, or the final product is slowly heated in an inert or oxygen-deficient environment at temperatures ranging from 200 to 300 °C [11,12 pp. 30-31]. The specialized literature describes three types of torrefaction: wet torrefaction [13], dry torrefaction [14] and ionic torrefaction [15]. Among these, dry torrefaction is the most widely used thermochemical treatment method for producing solid biofuels [16].

Regardless of the method used, torrefaction consists of several distinct stages: initial heating, pre-drying, post-drying, and intermediate heating [17]. However, the torrefaction mechanism remains a subject of scientific and practical interest, as it is specific to each type of biomass. It is important to note that research in this field varies significantly depending on the technological solutions applied [18,19]. In this context, studies focused on specific cases have been a constant concern for many researchers.

For example, researchers in Canada analysed the effects of torrefaction on rice and peanut husks, wood sawdust, and bagasse in a nitrogen environment, depending on exposure duration and temperature. It was found that biomass torrefied at 300 °C achieved the highest heating value (HHV = 25.68 MJ/kg), comparable to the HHV of lignin [20]. Kumar demonstrated that densifying biomass torrefied at 225 °C reduces energy consumption and improves densification productivity [21]. Additionally, the positive effects of pre-processing torrefaction on cereal residues were highlighted by [22] for corn and cotton stalks.

Researchers in Thailand studied the effects of torrefaction on four types of agricultural waste (corn cobs, coconut husks, cassava rhizomes, and rice husks) in an inert environment at temperatures ranging from 200 to 300 °C for 30 min. The results demonstrated that torrefaction is a promising technology for converting agricultural waste into solid biofuels, which can be used as an alternative to coal [23].

Information on the effects of torrefaction on different types of agricultural biomass is also available in studies by researchers from Poland and Sweden, who investigated wheat, rice, and rye husks and straw [24, 25], corn cobs, cotton, and sunflower stalks [26], corn and cotton stalks [27], as well as soybean husks, corn cobs, rice straw, and grapevine branches [28].

The torrefaction of corn residue pellets with wet combustion gases highlighted that, at high steam concentrations, the decomposition reaction of hemicellulose occurs more rapidly, with the optimal torrefaction conditions being an exposure temperature of 260 °C and a duration of 20 minutes [29]. The efficiency of water vapor presence in the torrefaction atmosphere is also demonstrated in [30], due to its influence on the degradation kinetics of solid biomass.

The effect of torrefaction on pellets produced from wheat straw and wood residues was analyzed using a laboratory setup that simulates heating conditions in the absence of oxygen. Torrefaction was conducted at temperatures of 240 - 270 °C for 15 min [31].

The results indicated that the calorific value of woody biomass increased by approximately 16%, confirming the efficiency of the torrefaction process in the production chain of agricultural residue pellets. This finding is also supported by the research of Tumuluru et al. [11,32].

The possibility of using residues from poppy (*Papaver somniferum* L.) and buckwheat (*Fagopyrum esculentum*) in the pyrolysis process to improve energy performance and utilize biomass waste for biochar production was analyzed by Saletnik et al. [33]. In this study, samples were maintained in a nitrogen atmosphere with 99.99% purity and a gas flow rate of 2 min^{-1} at temperatures of 400, 450, and 500 °C for 2 - 18 min.

Although there are numerous studies on the torrefaction of agricultural residues, the specific characteristics of torrefying pellets made from biomass generated by fruit shrubs have been less explored. The aim of this study is to estimate the effect of torrefaction on pellets produced from mixtures of fruit shrub residues.

The methodology used is based on a multifactorial study of the effects of torrefaction regimes on the quality of pellets produced from mixtures of sea buckthorn, blackberry, and wheat straw residues. The results obtained allowed for the optimization of the technological parameters of torrefaction.

2. Material and Methods

The research was conducted in the Solid Biofuels Scientific Laboratory of the Technical University of Moldova and in the Surface Engineering Laboratory within the Faculty of Mechanics at the "Gheorghe Asachi" Technical University of Iași. The study focused on two types of pellet samples:

1. Mixture of sea buckthorn plant residues (SBPR) 30% and blackberry plant residues (BPR) 70%, called SBPR+BPR.
2. Mixture of SBPR 30% and wheat straw (WS) 70%, called SBPR+WS.

The torrefaction of the pellets was carried out in a Nabertherm N41/H thermal treatment oven, equipped with a separate chamber and controlled atmosphere, allowing the simulation of the torrefaction process in an argon environment within a closed space (Figure 1).

The research was based on a 2^2 multifactorial experimental design with three levels. The considered influencing factors were the torrefaction temperature, ranging from 200 to 300 °C, and the exposure duration, between 10 and 30 min. Statistical data analysis was performed using STATGRAPHICS Centurion version 18 software.

For all the analyzed samples, the calorific value and ash content were determined. The gross calorific value was measured using the IKA C6000 isoperibol calorimeter, used in a constant volume environment, according to the SM EN ISO 18125:2017 standard. In this study, the calorific value is expressed as the net calorific value, adjusted to 10% moisture.

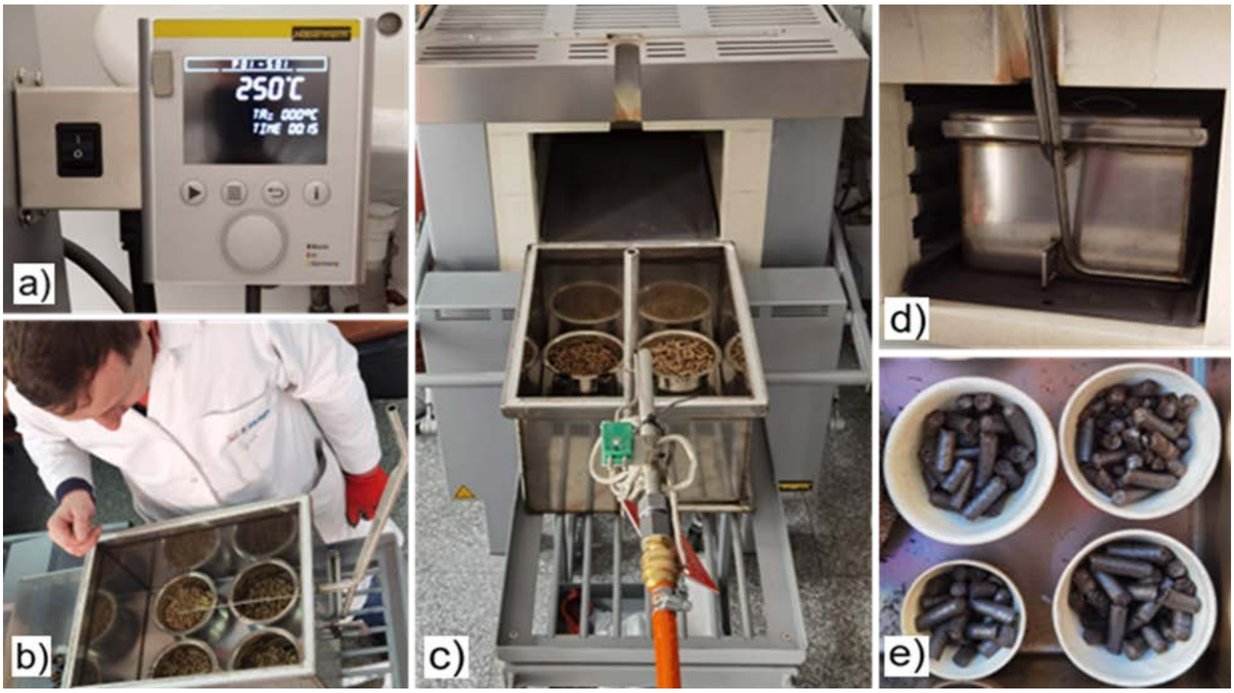

Figure 1. Sequences related to the torrefaction process of pellets from the Surface Engineering Laboratory at the Faculty of Mechanics, "Gheorghe Asachi" Technical University of Iași: a) electronic control panel for exposure time and temperature; b) and e) the samples investigated; c) Nabertherm N41/H thermal treatment oven before sample loading; d) protective box with gas inlet and outlet equipped with temperature sensors.

The ash content was determined according to the SM EN ISO 18122:2023 standard, using an LH 05/13 muffle furnace. The detailed procedure for determining the ash content is described in [4].

Before testing, the torrefied pellet samples were crushed using the Retsch SM 100 mill, equipped with a sieve with a mesh size of 1 mm. All tests were repeated five times, and the results were analyzed by determining the standard deviation and confidence interval.

3. Results and discussions

Following the statistical processing of the experimental data, the following regression equations were obtained, which adequately express the dependence of the examined factors on the torrefaction regimes:

$$Q = 6.46167+0.0471T+0.359583DE-0.000052T^2-0.000135T\cdot DE-0.00535DE^2 \qquad (1)$$

$$A = 0.67333+0.0108T+0.052833DE-0.00002T^2+0.00002T\cdot DE-0.00105DE^2, \qquad (2)$$

where: Q - calorific value; T - torrefaction temperature; DE - exposure duration; A - ash content.

The analysis of these equations allows for an understanding of the physico-chemical phenomena that occur during the torrefaction of the examined pellets.

Thus, the analysis of equation (1), the results of which are presented in Figure 2, shows that both the increase in torrefaction temperature and exposure duration lead to an increase in the net calorific value. At the same time, the second-order terms of the equation suggest a nonlinear behaviour, indicating that, at very high temperatures and durations, the calorific value begins to decrease. The interaction between temperature and exposure duration shows that their combined effects are not purely additive, meaning that, under certain conditions, they can negatively affect the efficiency of the process.

Analyzing the dynamics of the process (Figure 2), it is observed that the calorific value starts to increase from the first phase of the experiment. This can be explained by the fact that, in the temperature range of 200-250 °C, hemicellulose begins to degrade, releasing volatile compounds and improving the carbon-to-hydrogen ratio.

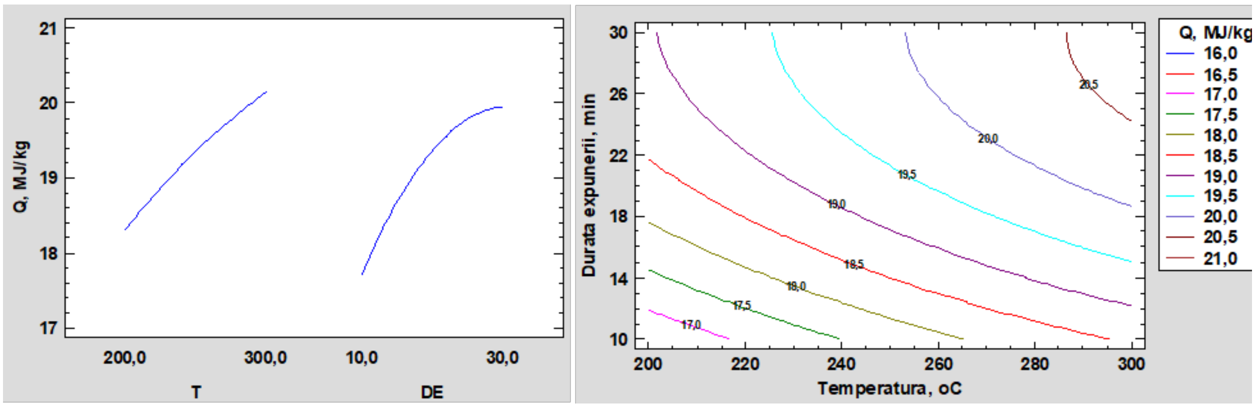

Figure 2. Diagram of the effects and response surface contours for the net calorific value (Q), adjusted to 10% moisture, of pellets obtained from SBPR+BPR, depending on the torrefaction temperature (T) and exposure duration (DE).

In the range of 250-300 °C, cellulose undergoes partial pyrolysis, leading to a reduction in mass and the formation of more carbonized structures. Lignin, due to its thermal stability, undergoes slower degradation, thus contributing to the increase in calorific value. However, at temperatures exceeding 300 °C, excessive mass loss occurs, resulting in a decrease in calorific value. When the temperature and exposure duration are too high, mass losses are amplified by the release of combustible gases (CO, CH_4, H_2), which negatively affect the energy efficiency of the final product. This phenomenon is reflected by the negative second-order terms of the regression equation.

These findings are consistent with the results obtained by the collaborators of the Scientific Laboratory of Solid Biofuels at UTM in studies on agricultural biomass, such as wheat straw and woody residues [31].

It is worth mentioning that the increase in torrefaction temperature and exposure duration leads to a slow increase in ash content (see equation 2 and Figure 3), indicating losses of organic mass. The second-order terms show that, after a certain threshold, the increase in temperature and duration may slow down the accumulation of ash.

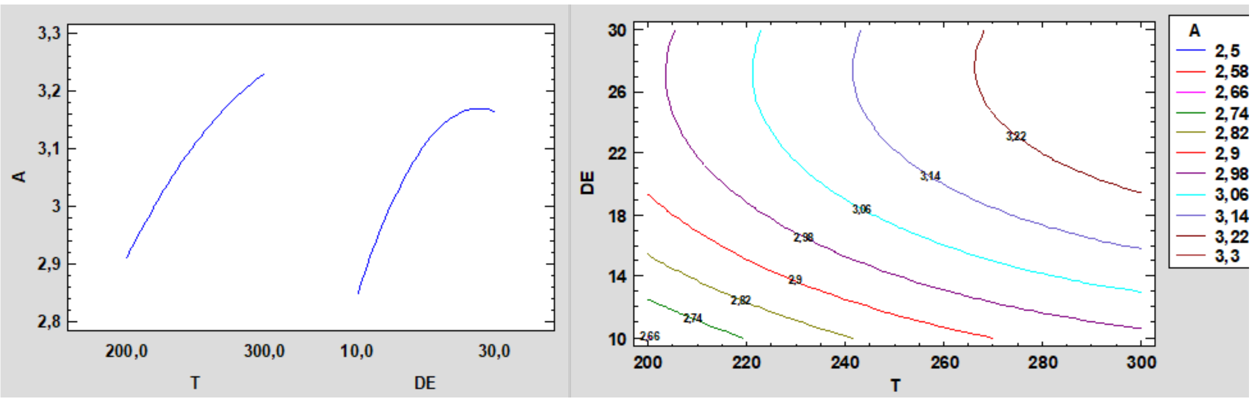

Figure 3. Diagram of the effects and response surface contours for the ash content (A) of pellets obtained from SBPR +BPR, depending on the torrefaction temperature (T) and exposure duration (DE).

The interaction between temperature and exposure duration is insignificant, suggesting that these two variables have an almost independent effect on the ash content. The increase in ash content can be explained by the fact that, as the temperature rises, volatile organic compounds are released, and the biomass becomes richer in fixed carbon and ash. At higher temperatures and longer durations, losses of organic matter accentuate the ratio between ash and total mass.

The negative higher-order terms suggest that, after a certain threshold, the increase in temperature no longer leads to a significant increase in ash content, as the mineral components reach a stable state.

Next, the changes in the net calorific value (Q) and ash content (A) of pellets obtained from SBPR + WS are presented, depending on the torrefaction temperature (T) and exposure duration (DE). The regression equations describing these dependencies are:

$$Q = 4.53333+0.05187T+0.34767DE - 0.00006T^2-0.00014T\cdot DE-0.0054DE^2 \qquad (3)$$

$$A = 4.45278+0.0112T+0.05458DE-0.00002T^2+0.00002T\cdot DE-0.00107DE^2 \qquad (4)$$

The Pareto diagram and the response surface contours (Figures 4 and 5) provide a clear visualization of the influence of temperature and exposure duration on the studied parameters.

The positive coefficients of the linear terms in regression equations (3) and (4) indicate that both the increase in temperature and exposure duration contribute to the initial increase in calorific value and ash content. At the same time, the negative coefficients in equation (3) suggest a nonlinear relationship, indicating that after a certain value, the increase in temperature and duration begins to have a negative effect on the calorific value. Additionally, from equation (4), it can be observed that after a certain threshold, the effect of increasing temperature and duration on ash content becomes less pronounced, as confirmed by the negative coefficients of the higher-order terms.

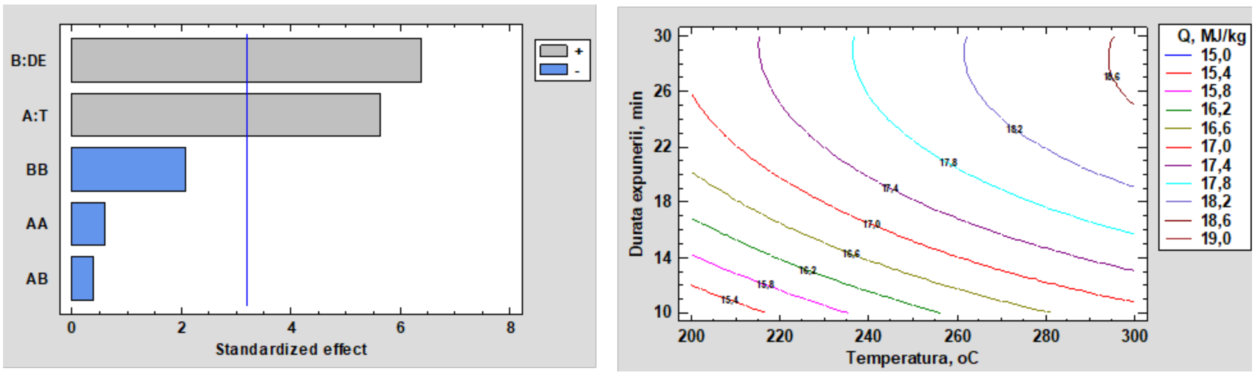

Figure 4. Pareto diagram and response surface contours for the net calorific value (Q), reported at 10% humidity of pellets obtained from SBPR +WS, depending on the torrefaction temperature (T) and exposure duration (DE).

It is also observed that the two parameters have an almost independent influence on the calorific value and ash content, and a simultaneous increase in both may insignificantly reduce the efficiency of the process.

The analysis of the diagrams in figures 2-5 highlights a high similarity between the dynamics of the torrefaction of SBPR +BPR pellets and that of SBPR +WS pellets, based on similar thermal processes. However, it should be noted that the torrefaction of mixtures containing wheat straw leads to a significant increase in ash content, reaching values over 7.14% for regimes that provide the highest calorific value of the pellets.

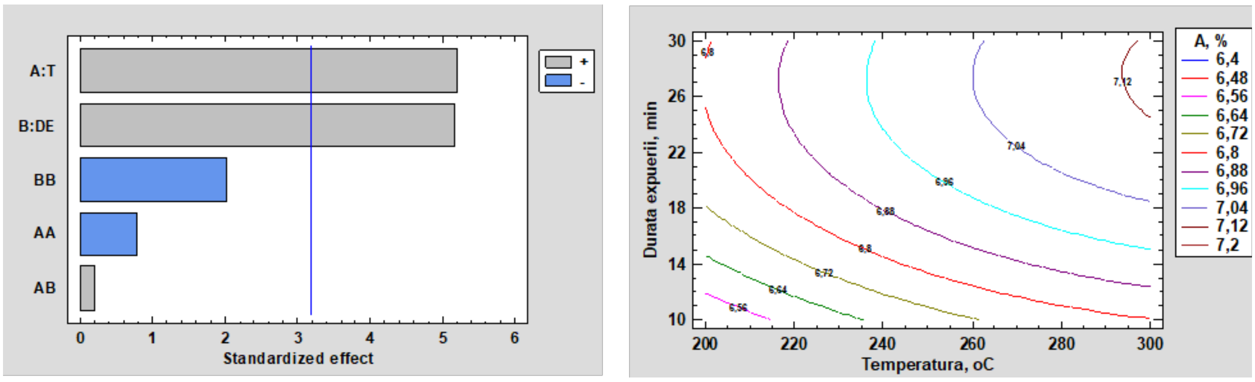

Figure 5. Pareto diagram and response surface contours for the ash content (A), reported at 10% humidity of pellets obtained from SBPR + WS, depending on the temperature and exposure duration (DE).

It is worth mentioning that the SM EN ISO 17225-6:2021 standard assigns this type of pellet to class B, which allows an ash content of up to 10%. To classify the pellets in category A, it is necessary to increase the proportion of SBPR or use other types of residues, while simultaneously reducing the percentage of WS.

5. Conclusions

Based on the findings, it can be concluded that:

1. The optimal torrefaction temperature for pellets made from SBPR + BPR is between 250 and 280 °C, where the calorific value is maximized and the increase in ash content is moderate.
2. The effective exposure duration for SBPR + BPR is between 15-25 min, as the increase in calorific value becomes insignificant after this point, while mass losses may become too large.
3. To prevent uncontrolled mass losses and a reduction in energy efficiency, temperatures above 300 °C and excessive durations (>30 min) should be avoided.
4. The obtained information can be used by solid biofuel producers to maximize the quality of pellets made from fruit shrub residues.

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