Juniperus waste wood as a source for obtaining pellets by single-screw extrusion method

A T Simitchiev¹, L V Lazarov², H N Fidan³, B V Bozadzhiev⁴ and A S Stoyanova²

¹ University of Food Technologies, Technical Faculty, Department of Machines and Apparatuses for Food and Biotechnological Industry, 26 Maritsa Blvd. 4000 Plovdiv, Bulgaria
² University of Food Technologies, Technological Faculty, Department of Tobacco, Sugar, Vegetable and Essential Oils, 26 Maritsa Blvd. 4000 Plovdiv, Bulgaria
³ University of Food Technologies, Faculty of Economics, Department of Nutrition and Tourism, 26 Maritsa Blvd. 4000 Plovdiv, Bulgaria
⁴ University of Food Technologies, Technological Faculty, Department of Cereals, Fodder, Bread and Confectionery Products, 26 Maritsa Blvd. 4000 Plovdiv, Bulgaria

e-mail: asimitchiev@gmail.com

Abstract. Waste wood (biomass) is one of the most valuable and multifunctional environmental, long term resources of Earth. The aim of this paper was to study the possibilities for obtaining pellets from Juniperus excelsa M. Bieb. biomass by a single-screw extrusion pre-treatment method. Biomass obtained after aqueous distillation of J. excelsa was used. The sample was grounded and sieved into dp ≤ 500 μm and dp > 500 μm particle in size. The experiments were carried out on a single-screw laboratory extruder “Brabender 20 DN”. According to our results, the pellets with dp ≤ 500 μm particle size were characterized with the following indicators: the mass flow rate (1.92 kg/h), sectional expansion index (1.03), specific mechanical energy (215.65 kJ/kg), density of the extrudates (0.44 g/cm³) and volumetric flow rate (4337.58 cm³/h). The samples obtained with dp > 500 μm particle size showed the following characteristics: the mass flow rate (1.30 kg/h), sectional expansion index (1.09), specific mechanical energy (535.23 kJ/kg), density of extrudates (0.48 g/cm³) and volumetric flow rate (2719.78 cm³/h). The biomass was a potential source for biofuel production. Pellets produced from biomass with a particle size dp > 500 μm had a higher density and sectional expansion index than those with a particle size dp ≤ 500 μm. However, their production was more economically impractical, which is due to their lower mass and volumetric flow rate, as well as higher specific mechanical energy.

1. Introduction
Waste wood (biomass) is one of the most valuable and multifunctional environmental, long term resources of Earth. It is used for the production of about 70% of the energy from renewable sources. It slowly decomposes with generating heat.

The principle of this natural process is used in the application of the plant biomass as a fuel source. The efficient combustion of plant biomass is a prerequisite for its use as an ecological biofuel. However, an important condition for maintaining the ecological balance is that no harmful emissions beyond limits must be generated during combustion.
A wide range of biomass has been used in the renewable energy industry such as firewood, energy wood chips, wood bark, wood pellets, wood briquettes, etc. [1], as their specifications were being described in various standards.

The physical characteristics of wood pellets, obtained from various biomass samples have been investigated by several authors [2-8].

Nunez et al. [2] determined the energy recovery of pellets from cork industrial waste. The results demonstrated that the cork pellets had a higher calorific value. Its physical characteristics were calorific value (20.62 MJ/kg), ash (2.52% – 2.82%), moisture (< 8%), bulk density (759 kg/m³), and durability (92.3% – 97.5%). The authors also reported that these indicators were related to the characteristics of pellets obtained from other types of biomass. For example, they were comparable with the indicators of pellets obtained with pine and eucalyptus wastes, where calorific value (16.53 – 18.13 MJ/kg), ash (0.59% – 2.69%), moisture (6.76% – 10.25%), bulk density (> 650 kg/m³), and durability (94% – 98%) were determined as well as pellets obtained with forest waste, where the calorific value (16.00 – 18.00 MJ/kg), ash (< 3%), moisture (2.79% – 7.46%), bulk density (> 650 kg/m³), durability (92% – 96%) indicators were reported.

Rabiskova et al. [3] investigated the influence of the extrusion die on pellet characteristics such as pellet size distributions, their mean diameter, density, hardness, friability, and repose angle. The formulation consisted of binary mixtures of theophylline monohydrate, which is a drug slightly soluble in water, and microcrystalline cellulose as an excipient. The reported pellet samples have shown low friability and excellent flow properties.

Repsa et al. [4] investigated the mechanical durability of briquettes and pellets of energy crop biomass. The briquettes were produced from common reed (Phragmites australis L.), reed canary grass (Phalaris arundinacea L.) and combined with peat. The results showed that mechanical durability depended on the peat quantity in the mixture.

Miranda et al. [5] studied pellets obtained from different biomass groups such as woody biomass (forest wastes - Pyrenean oak and Pyrenean sylvestris; wastes from wood industry – granulometric separation powder from cork industries and pine sawdust, and woody agricultural wastes – vine shoots and olive branches), herbaceous biomass (herbaceous agricultural wastes – barley straw and wheat straw), and fruit biomass (agro-industrial wastes – olive pomace and grape pomace). The authors investigated the moisture, bulk density, durability, chemical composition (C, H, N, and S), ash, and heating value. The results have shown considerable differences among the analyzed pellets, especially concerning ash content and N and S composition.

Ioelovich [6] investigated the thermodynamic properties (standard enthalpy, Gibbs free energy, and melting enthalpy of crystallites) of various allomorphs of cellulose.

Kazimirova et al. [7] determined the mechanical properties of pellets, which were obtained from ground poppy heads from the sieving of poppy seeds. The results have shown that the mechanical properties of pellets differed from each other. They showed lower values of the observed parameters in pellets obtained from dendromass.

Scatolino et al. [8] evaluated the possibility of lignocellulosic wastes of soybean culture, sugarcane bagasse, and eucalyptus wood for pellet production. The physical properties (moisture, bulk density, and unitary matrix), energetic properties (heating value and energetic density), chemical properties (volatiles, fixed carbon, and ash), and mechanical properties (mechanical durability and hardness) were determined.

In the flora of Bulgaria, different conifers (white and black pines, cedars, firs, and junipers) are used as raw materials mainly for the production of various aromatic products for application in medicine, cosmetics and perfumery. A very limited part of them is a source for the production of biofuels. There are five species of junipers in Bulgaria: J. communis L., J. oxycedrus L., J. pygmaea C. Koch, J. sabina L. and J. excelsa M. Bieb., as J. sabina and J. excelsa forests that are among the priority areas for plant conservation.

The aim of this paper was to study the possibilities for obtaining pellets from J. excelsa M. Bieb. biomass by a single-screw extrusion pre-treatment method.
2. Materials and methods

Biomass obtained after hydrodistillation of *J. excelsa* was used. The sample was grounded [9] and sieved into particle size $dp \leq 500 \mu m$ and $dp > 500 \mu m$ (Figure 1).

The moisture of the samples was 10.2% [10].

The experiments were carried out on a single-screw laboratory extruder “Brabender 20 DN” (Germany) with a working screw diameter of 20 mm, equipped with a measuring facility for torque Mn, N.m, the temperature of the material in the die and pressure inside the die.

![dp ≤ 500 μm][dp > 500 μm]

**Figure 1.** *J. excelsa* biomass.

All data were obtained directly from the control unit display of the extruder (Fig. 2).

![Extruder Diagram](image)

**Figure 2.** Single-screw laboratory extruder “Brabender 20 DN”.

1. Feeding device. 2. Screw 3. Cylinder 4. Die with nozzle and heater. 5. Heating devices.

Cylinder diameter 20.05 mm;
Cylinder length 406.5 mm;
L/D 20:1;
Cooling air, water.

Preliminary experiments were performed in order to establish the optimal extrusion regimes in which pellets with a clearly defined cylindrical structure and density were obtained. The complications created during these experiments were mainly related to the difficult flow of the product with particle size $dp > 500 \mu m$ through the feeding screw, as well as the complete decomposition of the obtained pellets.

The best results for the sample with $dp \leq 500 \mu m$ average particle size were observed under the following conditions: the internal diameter of the nozzle $D_o = 4$ mm; working screw with a compression ratio of 4:1; temperatures in the three zones of the extruder $t_1 = 120 ^\circ C$; $t_2 = 140 ^\circ C$; $t_3 = 160 ^\circ C$; the
speed of the feeding screw \( N_f = 20 \text{ min}^{-1} \); the speed of the working screw \( n = 220 \text{ min}^{-1} \). The following conditions for the sample with \( dp \geq 500 \mu m \) average particle size were used: the internal diameter of the nozzle \( D_o = 4 \text{ mm} \); working screw with a compression ratio of 4:1; temperatures in the three zones of the extruder \( t_1 = 120 \degree C \); \( t_2 = 140 \degree C \); \( t_3 = 160 \degree C \); the speed of the feeding screw \( N_f = 30 \text{ min}^{-1} \); the speed of the working screw \( n = 230 \text{ min}^{-1} \).

The obtained pellets are characterized by the following indicators:

1. Mass flow rate (kg/h):
   \[ Q_m = \frac{m}{t} \]  
   where: \( m \) – mass of biomass, kg; \( t \) – time, min.

2. Sectional expansion index:
   \[ SEI = \left( \frac{D_e}{D_0} \right) \]  
   where: \( D_e \) – the average diameter of the extrudate for ten samples, mm; \( D_0 \) – diameter of the die nozzle hole, mm.

3. Specific mechanical energy (kJ/kg):
   \[ SME = \frac{2.\pi.\frac{M_n.n}{60}.Q}{3.6} \]  
   where: \( M_n \) – torque, N.m. It is recorded from the display of the power supply unit of the laboratory extruder; \( n \) – the speed of the working screw, \( \text{min}^{-1} \); \( Q \) – mass flow rate, kg/h.

4. Density of pellets (g/cm\(^3\)):
   \[ \rho = \frac{M.(1-W/100)}{V-M.W/100} \]  
   where: \( M \) – the mass of biomass, g; \( V \) – sample volume, cm\(^3\); \( W \) – the moisture of biomass, %. The moisture of the products was determined by drying for 24 h at 105 \degree C [11].

5. Volumetric flow rate (cm\(^3\)/h):
   \[ Q_v = \frac{Q_m.\rho}{10^3} \]  

All experiments were performed at ambient temperature \( t_a = 23.5 \degree C \). Five parallel experiments were carried out in the aforementioned modes of operation, and the results are presented in the Results and discussion section.

3. Results and discussion

Physical parameters (sectional expansion index, mass flow rate, specific mechanical energy, volumetric flow rate, density and pressure) of pellets obtained by \( J. excelsa \) biomass were presented in Table 1.

| Sample | Sectional expansion index | Mass flow rate, kg/h | Specific mechanical energy, kJ/kg | Volumetric flow rate, cm\(^3\)/h | Density, g/cm\(^3\) | Pressure, MPa |
|--------|--------------------------|----------------------|-----------------------------------|----------------------------------|---------------------|-------------|
| > 500 \( \mu m \) | 1.09±0.03 | 1.30±0.01 | 535.23±2.95 | 2719.78±87.11 | 0.48±0.01 | 7.68±0.13 |
| \( \leq 500 \mu m \) | 1.03±0.02 | 1.92±0.06 | 215.65±10.21 | 4337.58±133.01 | 0.44±0.01 | 7.36±0.11 |

The data values are expressed as the mean ± SD (standard deviation (n=5)).

Our findings for pellets obtained from \( J. excelsa \) biomass differ from previous results reported in the literature [2, 3]. Since the moisture is crucial because it can affect the physicochemical parameters and
stability of the pellets, the differences between results obtained in this study and that reported in the literature could be explained both by the differences in the moisture of the different samples and the origin of the raw material.

Images of the pellets obtained from *J. excelsa* biomass with two-particle sizes were shown (Figure 3a and Figure 3b). The presence of small dust particles was visible, which was an indicator of crumbliness and low density of the pellets. Expansion is an important indicator of food products obtained by forming with high temperatures and low moisture. The sectional expansion index usually depends on the composition of the product as well as the extrusion modes. The high values of the torque, pressure and temperature in the extruder help to convert the processed products into viscoelastic melts. This transformation depends on the moisture of the extruded product and the extrusion modes.

The sectional expansion index is an important indicator determining the texture and hardness of the final product and giving important information about the quality of the extrudate [12].

![Images of pellets from J. excelsa biomass.](image)

The expansion index of the obtained pellets represents their extension when they exit through the nozzle. The sectional expansion index of pellets with dp ≤ 500 μm particle size was lower compared to those obtained with larger particle sizes. This could be due to the weaker compression of the product during extrusion, as could also be seen from the density results, which could be explained by the differences in the cellulose content [13]. The smaller particle size led to the presence of less pronounced microcracks in the structure of the final product. These microcracks affect the expansion of the final product due to the air entering into the microcracks. The results were supported with the pressure during extrusion, which was lower for the sample with particle size dp ≤ 500 μm than that obtained for sample with dp > 500 μm. The pressure can be controlled during the extrusion process. The modes that could be applied were changing the speed of the working screw, the degree of compression of the screw used, and the diameter of the nozzle through which the product exits. Preliminary experiments were carried out in order to increase the pressure and all of them showed that the most optimal results could be obtained with the extruder operating with modes described above.

Density is an important indicator that characterizes the structural and mechanical changes in the product. The pellets were crumbly and with low density, which could be due to the fibrous nature of the processed product. It was necessary to add a material that better gather the particles of the final product in order to obtain pellets with a higher density and expansion index [9, 10]. Another way to increase the density and strength of the pellets was to use a specialized granulator for fibrous materials. This would increase the pressure and density during pressuring. According to our results, a higher density of the sample with particle size dp > 500 μm was obtained. This could be due to the larger particle size of the biomass, as well as the higher pressure used during extrusion.

The volumetric flow rate of the extrudates is an indicator influencing the volume of production as well as productivity. The volumetric flow rate of pellets produced with a smaller particle size (dp ≤ 500 μm) was determined over 37% higher than that observed at dp > 500 μm. The extrusion rate of the
pellets from biomass with a smaller particle size was significantly higher than that of the other particle fraction. The velocity affected the mass flow but an inverse relationship between the volumetric flow rate and the sectional expansion index was determined. Therefore, the higher volume of production did not lead to higher quality pellets during the extruding of fibrous materials. The low density and expansion index contributed to the easier disintegration of the pellets during their cutting and further expedition. Productivity (mass flow rate) and specific energy consumption were also calculated. Productivity (expressed by mass flow rate) is a basic technical and economic parameter that characterizes the operation of the extruder. The productivity of single-screw extruders is influenced by the speed of the screw, the pressure, and the construction of the die [14].

The mass flow rate of samples with a dp ≤ 500μm particle size was 22% higher, the reason for which could be the higher extrusion speed and the possibility for better filling of the inter-turn space of the screw by the product with smaller particle size. Backflow often occurs while moving through the inter-turn space of the screw in the samples obtained with larger particle sizes. It was because of the excessive filling of this space and the inability of the screw to transport and compress the raw material. Moreover, larger particles increased friction and the torque between the screw and the extruder housing, which had a negative effect on both speed and mass flow rate.

Energy consumption is a key indicator of any mechanical process in any industry. The amount of energy consumed affects the degree of macromolecular transformations and interactions that occur during the extrusion process, such as the degree of gelatinization of polysaccharides, the rheological properties of the molten material, etc. [15, 16]. In the present study, the extrusion of particles with dp ≤ 500 μm led to lower energy consumption. This was mainly due to higher productivity and lower torque during operation.

4. Conclusions
The J. excelsa biomass was a residual and waste product obtained during the essential oil production. The biomass was a potential source for biofuel production. Pellets produced from biomass with a particle size dp > 500 μm had a higher density and sectional expansion index than those with a particle size dp ≤ 500 μm. However, their production was more economically unfeasible due to their lower mass flow rate and volumetric flow rate, as well as higher specific energy consumption.

Acknowledgements
The authors acknowledge the support by the National Science Fund of Bulgaria, project No KPI-06-H36/14.

References
[1] Sabeva G, Zvetkov N, Boycheva S, Gadjanov P and Stankov N 2012 Science Conference EFM (Sozopol) 63-70
[2] Nunes L, Maria's J and Catalao J 2013 Fuel 113 24-30
[3] Rabiskova M, Weingartova D and Haring A 2007 Ceska a Slovenska Farmacie 56 17-20
[4] Repsa E, Kronberg E and Pudans E 2014 Engineering for Rural Development (Jelgava) 436-439
[5] Miranda T, Montero I, Sepulveda F, Arranz J, Rojas C and Nogales S 2015 Materials 8 1413-1427
[6] Ioelovich M. 2019 Chem Press. 9 259-265
[7] Kazimirova V, Kubik L, Chrastima J and Gietl T 2017 Agronomy Research 15 1906-1917
[8] Scatolino M, Neto L, Protaslo T, Carneiro A, Andrade C, Guimaraes Junior J and Mended L Wasre Biomass Valor doi: 10.1007/s12649-017-0010-2
[9] Obidzinski S 2014 International Agrophysics 28 85-91
[10] Gil M, Oulego P, Casual M, Pevida C, Pis J-J and Riviera F 2010 Bioresource Technology 101 8859-8867
[11] BDS EN ISO 712:2010
[12] Patil R P, Berrios J, Tang J and Swanson B 2007 American society of Agricultural and Biological
[13] Lazarov L, Bozadzhiev B, Simitchiev A and Stoyanova A 2020 *Youth Forum Science, Technology, Innovation, Business – 2020* (Plovdiv) 42-47
[14] Rosentrater K, Muthukumarappan K and Kannadhason S 2009 *Journal of Aquaculture Feed Science and Nutrition* 1 22-38
[15] Petrova T, Nenov V and Chorbanov B 2007 *UFT Scientific Papers* (in Bulgarian) 54 160-165
[16] Toshkov H 2011 *Study of the extrusion process of commercial fish species* PhD Thesis UFT Plovdiv (in Bulgarian)