

Article

The Use of Black Pine Bark for Improving the Properties of Wood Pellets

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Abstract: The requirement for alternative raw materials for fuel pellets that would enable the use of readily available low-cost renewable resources and waste materials, such as bark, has always attracted interest. The aim of the current work was to assess the effect of black pine (*Pinus nigra* L.) bark content (0%–100%) as well as densification temperature on the properties of black pine wood pellets produced in a single pellet die. The quality assessment of the pellets was carried out by the determination of radial compression strength, density, moisture content, ash content, and surface roughness. The results showed that adding black pine bark to the pellet feedstock resulted in the production of substantially smoother and moderately denser pellets, which also exhibited higher mechanical strength than that of the respective pellets of pure wood. Finally, it was shown that black pine bark can be a valuable raw material, which can induce improved bonding of biomass particles and may provide the opportunity to create pellets of favorable characteristics at a lower temperature compared to those made of pure wood.

Keywords: bark; black pine; compression strength; densification; pellet; roughness; wood



Citation: Lykidis, C.; Kamperidou, V.; Mantanis, G.I. The Use of Black Pine Bark for Improving the Properties of Wood Pellets. *Forests* **2023**, *14*, 1069. <https://doi.org/10.3390/f14061069>

Academic Editor: Yongfeng Luo

Received: 25 April 2023

Revised: 18 May 2023

Accepted: 21 May 2023

Published: 23 May 2023



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1. Introduction

The feedstock used for wood pellets usually includes low-quality virgin wood, small-diameter logs of irregular wood structure, logging, and processing residues. The valorization of such underutilized waste biomass material is gaining interest for various applications. In this context, it would be beneficial to search for alternative low-cost raw materials that have not been extensively utilized so far [1] in order to meet the increasing raw material requirements of the pellet industry, especially given the concurrent high demands of the paper industry and particleboard/fiberboard/pulping industry for the same raw materials.

The need to transform biomass into a dense energy carrier, such as pellets, emerges from the fact that in its densified form, wooden biomass presents lower volume, consistent quality and higher energy density properties which facilitate affordable transportation and storage as well as the lower generation of dust/fines and risk of explosions, lending to its use as a feedstock in less challenging and expensive energy conversion systems.

Among the most crucial quality factors that should be taken into account when producing biomass pellets is the mechanical durability. Mechanical durability refers to pellets' capacity to tolerate pressure, friction, and shock during handling and transportation, and constitutes an important parameter that highly affects the proper transport and combustion of biomass [2,3]. According to standard ISO 17831-1, pellets characterized by mechanical durability higher than 97.5% are regarded as appropriate for residential application. Durability is based on strong adhesion forces between particles, which have been attributed to appropriate densification temperatures and pressure, raw material crystallization, chemical

reaction, and adhesive or lignin hardening following densification [4,5]. Specifically, mechanical durability could be affected by parameters related to the raw material (chemical composition, moisture content, particle size, and formulation) as well as the pelletizing process (heating time, pressure time, post-treatments, etc.) [6]. Under the conditions drawn up during the densification process, lignin (21%–32% of the overall mass) presents glass transition while extractives (approximately 2%–25%), such as starch and sugars, could polymerize acting as natural binding agents [7–9]. Furthermore, cellulose crystallinity may increase, resulting in the further enhancement of the inter-particle adhesion [10,11], while hemicelluloses may also have a positive effect on biomass pelletization [12]. In addition, Stelte et al. [13] have reported that wood extractives may reduce the compressive strength of pellets, while Telmo and Lousada [14] reported their high contribution to the increase in calorific value.

Various additives have been utilized to enhance the bonding of biomass materials including brown sugar powder [15], proteinaceous waste [16], bio-oil [17], microalgae [18], rapeseed flour, coffee meal, pinecones, lignin powder [19], miscanthus, potato starch [20], cornstarch and molasses [21], and citrus peels [22]. However, only a few studies have been published on the impact of bark particles on the properties of wood pellets, and research into the development of pellets using bark proportions is still in its early stages [23,24].

Annually, the amount of bark synthesized varies between 179.557.100 and 359.114.200 m³ [25]. It is a readily available, low-cost, biological, and renewable raw material with valuable properties [26]. Nevertheless, bark is difficult to be utilized in paper and chipboard industries due to its high extractives content, dark color, and lower mechanical strength. Additionally, the high ash, nitrogen, and sulfur content compared to wood [8,23] constitutes a limiting factor with regard to its energy utilization, especially in small-scale burning systems where problems related to high ash content are difficult to manage [1]. Vinterback [7] has reported that purely bark-based pellets may cause the ash components to sinter on the burner walls, which would not occur in the case of wood–bark mixture-based pellets.

Bark extractives, mainly tannins, exhibit adhesive properties [27]; wood bonding without any binder is technically feasible even through bark self-bonding is attributed mainly to the development of physical bonds and the polymerization of bark extractives under suitable conditions of temperature, pressure, and time [28,29]. Self-bonding can be facilitated by high temperatures since they induce the thermal–chemical degradation of bark and the production of compounds that are ready to react with bark extractives and lignin [30]. Moreover, the granulometry and the share of bark particles are critical factors affecting self-bonding [29].

Szyszlak-Bargłowicz et al. [31] reported the low calorific value of bark pellets due to the high ash content and loss of volatiles during drying, while other researchers [10,32–35] reported higher calorific values for bark than for wood. Generally, the calorific value of dry bark amounts to 17,000–22,000 kJ kg⁻¹ (4.7–6.1 kWhkg⁻¹) and is comparable to that of dry wood [36]. Kamperidou et al. [35] reported that the incorporation of bark of some fast-growing hardwood species (pseudoacacia, poplar, ailanthus, and paulownia) to the feedstock of the same species of wood material significantly increased the calorific value of the produced biofuels and that optimized wood–bark ratios could result in biofuels of low ash content and adequate quality for both commercial (residential and industrial) applications.

Bark pellets produced in lab-scale experiments have demonstrated satisfying mechanical durability [1,7,31]. A study of the effect of pine bark on the quality of pellets containing 0, 5, 10, 30, and 100% bark showed that among the tested combinations, pellets made of 100% bark demonstrated the highest mechanical durability. Wistara et al. [6] reported similar results with palm tree bark and wood. A bark proportion of 10% has been found to be optimal since it corresponds to an improvement of important technical properties without exceeding the ash content requirement (less than 0.7%) [1,23,37]. Lehtikangas [10] reported the excellent resistance of bark pellets to moisture fluctuations and dimensional changes, as well as higher density, compared to those made of wood raw material.

Surface roughness has been acknowledged as a quality indicator of pellets, although it still has not been extensively studied [8,38–40]. Within the above context, it would be interesting to assess the effects of bark presence on the quality of wood pellets in terms of their density and mechanical durability, as well as on the surface roughness, as this could provide a potential tool for the in-line non-destructive assessment of pellet quality within the production process or as a selection criterion prior to consumption. Therefore, the aim of the current study was to determine the effects of bark content, as well as that of densification temperature, on the physical and mechanical properties of wood pellets made of black pine wood.

2. Materials and Methods

For the purposes of this research, a 25-year black pine (*Pinus nigra* L.) trunk that originated from a Greek forest (Pindos region) was used. Wood and bark were manually separated. Representative, defect-free wood specimens were sampled from several different regions of the trunk (different heights from 30–130 cm and different areas in the direction from pith to cambium zone). Wood and bark were crushed using a hammer mill (Laizhou Chengda Machinery Co., Ltd., Yantai, China) and sieved to acquire particle dimensions of 0.5–1 mm. Afterward, the materials were conditioned in a climate chamber at 20 ± 0.1 °C, $65 \pm 1\%$ RH until constant weight. Prior to densification, the mean moisture content of the used materials was determined according to EN14774-3:2009 [41] and was found to be 11.22% for wood and 12.99% for bark. The ash content of the materials was determined according to ASTM D1102-2001 [42] averaging four replicates. Specifically, samples of at least 1 g were weighed to the nearest 0.1 mg in dry, clean, and pre-weighed porcelain crucibles and then transferred to a cold muffle furnace (Heraeus MR 170, Leipzig, Germany) with a ventilation rate of about 5 changes per minute. The samples were heated to 250 °C within 50 min and the temperature was kept constant for 60 min. In the next step, the temperature was increased to 580 °C within 60 min and was maintained at that level for 2 h. Afterwards, the crucibles were transferred to an empty desiccator without a lid for 5 min, followed by 15 min with a closed lid, and then weighed.

In order to study the effect of bark content on the quality of wood pellets, various bark to wood mixtures (w:w) were prepared, namely 0:100, 10:90, 30:70, 60:40, and 100:0, respectively. The mixtures were manually homogenized for approximately 10 min/mixture. The above materials were used for the production of pellets using a laboratory-scale single die press (Figure 1) with a diameter of 12 mm and adapted to an Amsler hydraulic universal testing machine with a capacity of 4 t. The use of a single pellet die has been also used elsewhere for the production of pellets under controlled production parameters [43–45]. In this research, the used pellet press was used at a closing speed of 10 mm/min and temperatures of 80, 100, 120, 140, and 160 °C. The densification load was 4 t.

Immediately after the production of each pellet, its diameter, thickness, and weight were measured using a digital caliper with a resolution of 0.01 mm (Mitutoyo 500-196-30) and a balance with a resolution of 0.001 mg. Using the above measurements, the pellet density was also calculated and the results were expressed in kilograms per cubic meter (kg/m^3). The produced pellets were conditioned in a climate chamber at 20 ± 0.1 °C, $65 \pm 1\%$ RH and their MC was determined according to EN ISO 18134-1 [46]. After conditioning, the dimensions (diameter, thickness, and weight) were measured again in order to assess any dimensional changes after the conditioning.

Mechanical durability is considered one of the most important parameters for assessing pellet quality since it is the optimal indication of the risk of pellet deterioration and fines production during transport and handling [23]. In the case of single pellet production, mechanical durability is usually indirectly assessed throughout the determination of the quasi-static radial compression strength of pellets [47]. In this research, the radial compression strength was determined using the above-mentioned testing machine (Figure 2). Up to 10 pellets for each variable were tested. Each pellet was radially loaded under constant velocity of the loading head and the maximum compressive load was recorded for each test.



Figure 1. Single die press fitted to a Universal Testing Machine.



Figure 2. (A) Mechanical durability test of 100% bark pellet and (B) destroyed pellet samples after the completion of the testing process.

The surface roughness of all produced pellets was determined using a Mitutoyo SurfTest SJ-301 stylus-type profilometer according to ISO 21920-2:2021 [48]. The measurement speed, pin diameter, and top angle of the pin tool were 10 mm/min, 4 μ m, and 90°, respectively. The sampling length was 1.8 mm. For each specimen, 4 measurements were carried out along the thickness of the specimens on the curved surfaces of cylinder-shaped pellets (Figure 3) and were averaged. Among the typically determined roughness parameters, the mean arithmetic deviation of profile (R_a) is considered the most appropriate for similar materials and for this reason, it was used in this study [49]. Prior to the measurements, the apparatus was calibrated using a Mitutoyo 178-601 surface roughness standard protocol. All roughness measurements were carried out after careful conditioning of the specimens at 20 ± 0.1 °C and $65 \pm 1\%$ RH.

Statistical analysis was carried out using SPSS Statistics, which was used to determine statistically significant differences between the property values of the prepared pellets of the different categories, while the 3D graphs were created by using *Statistica*.

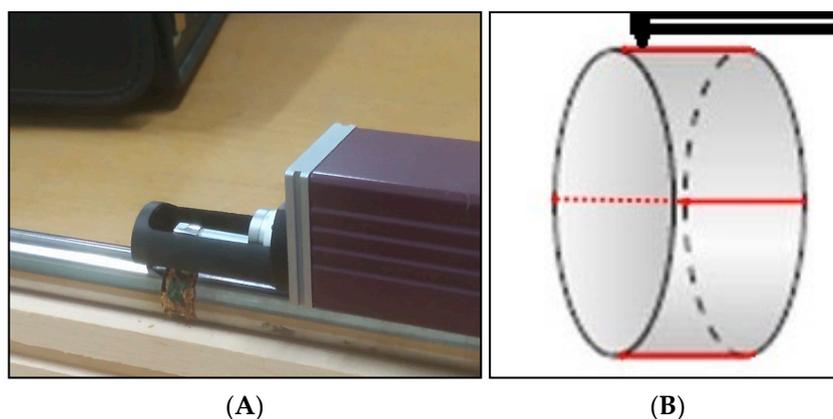


Figure 3. (A) Anchoring technique of the pellet-sample prior to the roughness measurement procedure and (B) the foursurface roughness measurement longitudinal paths on the pellet sample curved surfaces.

3. Results and Discussion

According to the results (Table 1 and Figure 4), the density of pellets seems to increase upon increase in the bark content. This increase seems to be enhanced by the simultaneous increase in temperature in the range of 80 to 160 °C. Therefore, the bark material seems to have high potential for the manufacture of higher density biomass materials (solid biofuels, bio-based products, etc.). Specifically, among the studied material types, pellets made of 100% bark under a densification temperature of 160 °C demonstrated the highest density, while the lowest density values were recorded by pellets made of pure wood at the lowest temperatures (80 and 100 °C). Increased density in pelletized materials usually corresponds to a high number of particles that are in close proximity to one another. The bark’s components, including tannins, hemicelluloses, lignin, and cellulose nanofibers, are those contributing to the closer proximity and stronger adhesion between the particles [50]. Furthermore, Lehtikangas [10] reported that bark is more sensitive to temperature and pressure than wood because of the comparatively large amount of amorphous chemicals, lignin, and extractives contributing to higher pelletized materials densities. It should also be considered that bark usually has slightly higher moisture content than wood due to the differences in chemical composition of these materials (in this study, the moisture contents of black pine wood and bark were 11.22% and 12.99%, respectively) and it is well accepted that moisture improves binding of pelletized materials [51–53] due to its influence on the glass transition temperature of lignin.

Table 1. Properties of pellets made from different material categories (mean values, standard deviation values—SD, and the number of examined specimens—N).

Temperature (°C)		80					100					120					140					160									
Bark Content (%)		0	20	40	60	80	100	0	20	40	60	80	100	0	20	40	60	80	100	0	20	40	60	80	100	0	20	40	60	80	100
Compression strength (kg)	mean	5.73	4.74	5.75	6.13	6.82	7.33	6.33	4.68	4.78	5.52	5.98	6.65	9.09	9.66	10.58	7.69	6.80	6.57	8.45	9.93	10.08	8.53	10.20							
	N	12	10	13	6	6	6	9	12	11	6	4	6	10	11	4	8	4	6	11	9	5	12	5							
	SD	1.11	0.68	1.39	1.23	1.10	1.54	1.22	1.99	1.00	1.45	0.62	1.92	1.73	0.88	1.24	1.98	2.03	1.20	1.87	2.24	2.61	1.68	3.96							
Roughness R_a (μm)	mean	3.997	3.508	2.185	2.476	1.952	1.559	3.183	2.741	2.179	1.990	1.728	1.497	3.684	2.265	2.036	1.727	1.489	1.493	4.283	3.214	2.568	4.384	2.868							
	N	20	20	21	10	9	10	20	20	18	10	7	9	21	20	9	12	7	10	20	20	10	20	9							
	SD	0.919	0.660	0.330	0.354	0.351	0.221	0.701	0.642	0.425	0.422	0.177	0.191	0.508	0.461	0.261	0.163	0.251	0.129	0.854	0.513	0.414	0.664	0.398							
Density (kg/m^3)	mean	946	962	1002	998	1014	1077	903	934	990	983	1028	1016	955	1028	1023	1051	1074	1109	930	1013	1055	926	1008							
	N	20	20	21	10	10	10	20	20	18	10	7	10	21	20	9	12	7	10	20	20	10	20	9							
	SD	42.4	62.2	31.7	21.5	31.6	38.9	44.0	58.6	68.6	62.5	35.8	39.8	34.8	35.9	23.5	42.0	59.6	33.0	63.1	49.2	55.0	33.8	43.8							

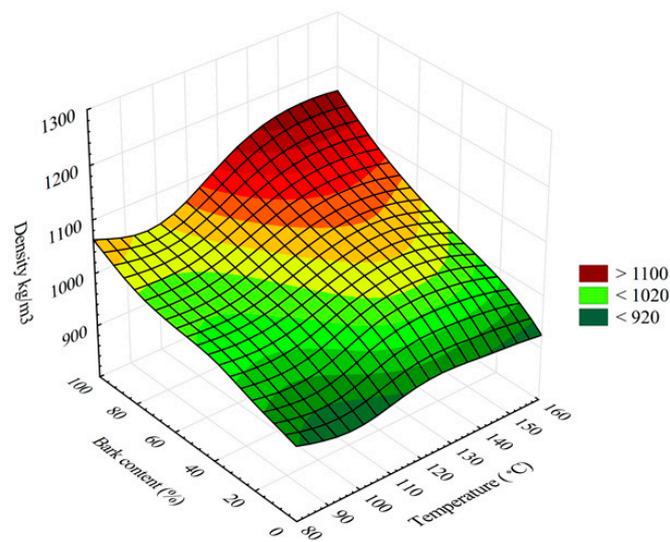


Figure 4. Density against bark content and densification temperature of the produced pellets (distance weighted least squares).

The radial compression strength of the produced pellets (Table 1 and Figure 5) was positively affected by the presence of bark. The increase in the bark ratio up to 40% resulted in higher radial compression strength, though further increase seems to contribute to a deterioration of pellet strength. This result complies with the results published by Wistara et al. [6] who reported that the highest pellet durability resulted from densification at 130 °C and a bark ratio of 30%. Additionally, Lehtikangas [10] reported that pellets made of bark demonstrated the highest durability, whereas sawdust pellets showed the lowest one, but without statistically significant differences among them. Terzopoulou et al. [52] reported that an increase in the bark content from 0% to 7% in pellets feedstock material had a beneficial effect on the mechanical durability of cypress species (*Cupressus arizonica* and *Cupressus sempervirens*) fuel pellets of conventional dimensions, which corresponds to a mechanical durability improvement that ranges between 1.62 and 2.3%. Lerma-Arce et al. [54] also confirmed that an increase in the bark content increases the mechanical durability of pellets. This increase could be attributed to the higher lignin and extractives concentration of bark compared to wood material exhibiting a positive effect on the cohesion mechanism during the densification process [10,55].

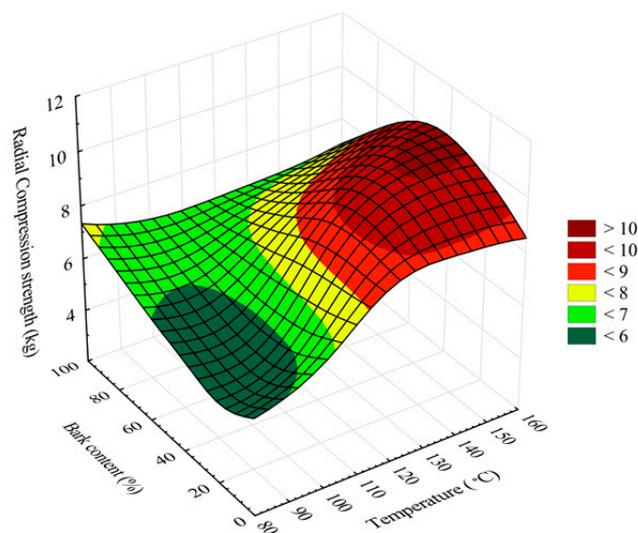


Figure 5. Radial compression strength against bark content and densification temperature of the produced pellets (distance weighted least squares).

Consequently, these essential findings of previous and current research work reveal a high potential for bark utilization in solid biofuels and other densified biomass materials. Additionally, bark presence in pellet feedstock could provide the opportunity for pellets of satisfying properties to be produced at lower temperatures than those made of pure wood, in this way reducing the required energy consumption and production cost.

Additionally, densification temperature seems the most important factor influencing mechanical durability. Densification temperatures up to 100 °C resulted in the lowest radial compression strength values among the tested variables without statistically significant differences between 80 °C and 100 °C. An increase in temperature to 120 °C induced improved bonding, which was evident by the increased radial compression strength of the produced pellets. Nevertheless, further increases at the level of 140 °C and 160 °C did not correspond to an additional increase in radial strength. This indicates that the beneficial effect of temperatures up to 120 °C upon softening of the densified materials is probably balanced by the thermal degradation of the material that occurs at higher temperatures. The level of pressure applied during densification is a factor of significant influence on the mechanical durability of the pellets [54]; however, in the present study, the pressure applied during densification of all pellets was constant (4 tons) in order to allow the other factors to be compared (densification temperature and bark percentage). According to previous studies, the mechanical durability of pellets appears to be strongly correlated to their density [38,52]. Nevertheless, in this research, the linear correlation between these two variables was not found to be strong (Figure 6).

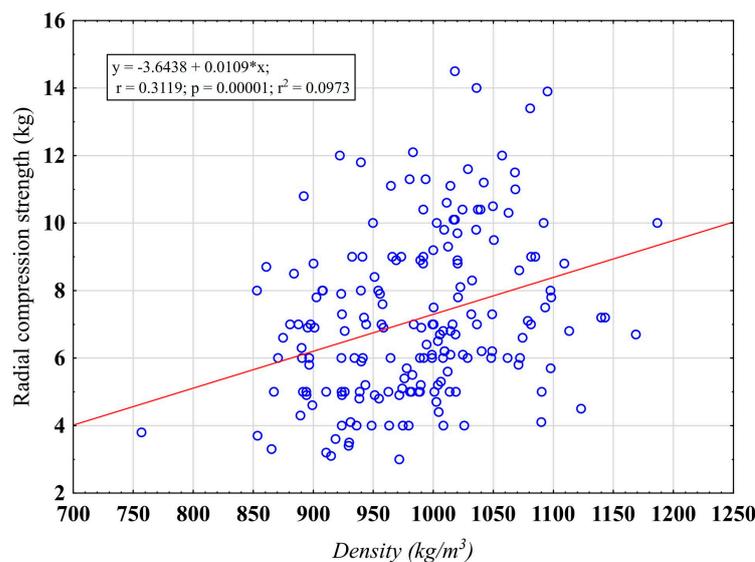


Figure 6. Density against radial compression strength values of the produced pellets.

Regarding the surface roughness of pellets, the use of black pine bark resulted in R_a mean and standard deviation decrease (Table 1 and Figure 7). The highest mean roughness value was presented by pellets made of 100% wood material, whereas the lowest value was recorded by pellets made of 100% bark. Furthermore, the effect of the densification temperature factor on pellet roughness seems to be less strong than bark content, though still there is an effect that corresponds to the respective effects regarding radial compression strength: the increase in the densification temperature up to 120 °C resulted in the decrease in roughness, while a further increase to levels higher than 120 °C resulted in an increase in pellet roughness. This fact indicates that the lower temperatures (below 120 °C) are not high enough to trigger various beneficial chemical reactions on the surface of the densified products, such as migration of extractives, surface modification, and glass transition of lignin, which would result in a smooth surface. Accordingly, higher temperatures (above 120 °C) are likely to exceed the range of positive impact of heat, since thermal degradation/oxidation, etc., reactions may take place, leading to

an increase in the surface porosity of the pellet and resulting increased roughness. A thorough characterization of the chemical composition in the surface layer of pellets, as well as a structural analysis, would probably contribute to the interpretation of the abovementioned results.

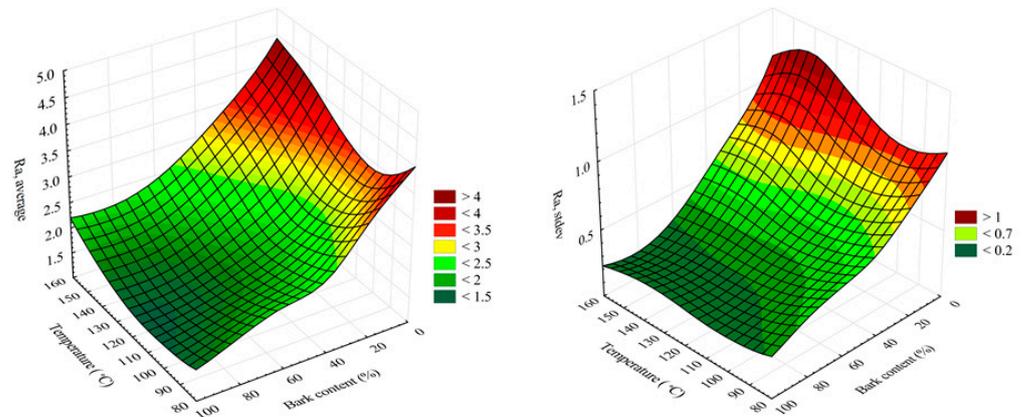


Figure 7. R_a (left: average and right: standard deviation) against bark content and densification temperature of the produced pellets (distance weighted least squares).

The above results regarding mean R_a values are further validated by the respective standard deviation values of the same parameter (Figure 6, right). The increase in the bark content, as well as temperature, results in a more homogeneous surface in terms of roughness. The few relevant studies in the literature reveal that the high roughness of pellet surface is correlated with lower quality of wooden pellets, more specifically to lower mechanical durability, higher ash content, and lower calorific values, at least for fuel pellets sold in the market [8]. Despite the above results regarding the clear positive effect of bark content on the radial compression strength as well as surface smoothness of produced pellets, Figure 7 as well as Figure 8 suggest that there is no clear correlation between surface roughness and the mechanical durability of pellets. This finding can lead to the conclusion that the mechanical properties of pellets are a result of multiple factors that should be systematically assessed, in order to deeply understand the fundamentals of the bonding mechanisms during densification. Furthermore, the weak correlation of R_a against the density values of the produced pellets (Figure 9) validates the above conclusion.

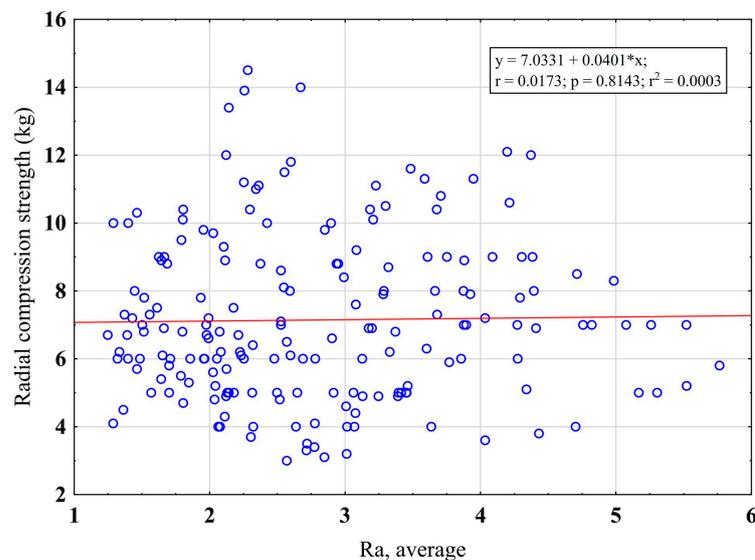


Figure 8. Average R_a values against the radial compression strength of the produced pellets.

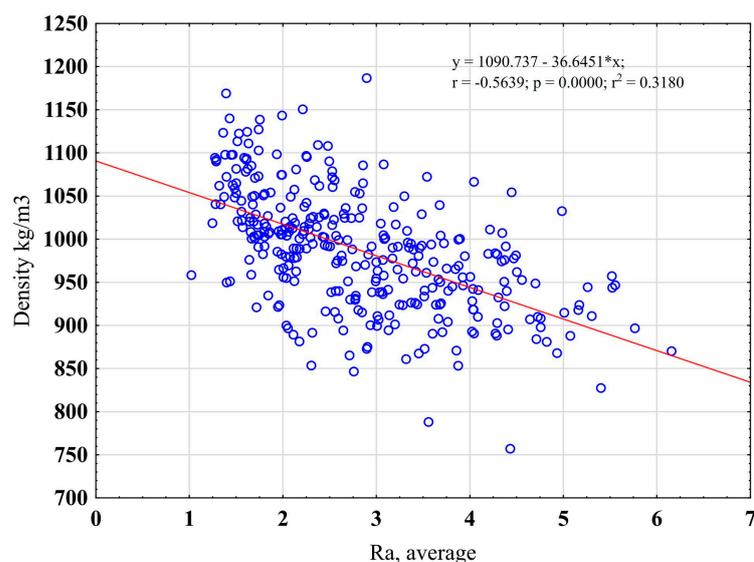


Figure 9. Average Ra values against the density of the produced pellets.

According to the findings of the current study, the ash content of wood was 0.24%, while the corresponding value of bark was 5.21%. Furthermore, the mixtures of bark and wood materials exhibited corresponding ash content values, as expected (Table 2). Bark content and ash content seem to be strongly correlated.

Table 2. Mean ash content values of the considered material categories (bark:wood ratios).

Bark:WoodRatio	0:100	20:80	40:60	60:40	80:20	100:0
Ash content (%)	0.24	1.24	2.23	3.22	4.21	5.21

According to EN-Plus (ISO 18122), the ash content threshold values for A1, A2, and B classes of pellets intended for residential use are 0.7%, 1.2%, and 2%, respectively. Pellets approved for industrial uses are certified as class I (industrial grade) and the respective threshold value of ash content is 3%. In this context, only the pellets that are purely made of black pine wood or those of low (up to 11.3%) bark content could be used as feedstock for the production of premium quality class (A1) pellets. Pellets made of up to 20% bark could be classified as A2; to fulfill the requirement for class B, bark contents should be in the range of 20%–36%. Regarding industrial use, the bark content could be in the range of 36%–56%, while bark ratios higher than 56% cannot be used as feedstock material in the production of solid biofuels, though could be used in the production of other densified biomass products, in applications where ash content is not a matter of concern.

In addition, Lerma-Arce et al. [54] reported that it is feasible to obtain high-quality pellets, in terms of ash content, as well as mechanical durability, bulk density, calorific value, moisture content, etc., from barked logs and branches of pine species, specifically of *Pinus halepensis* and *Pinus pinaster*, in which bark contents of 8.76% and 11.43% were recorded, respectively.

The findings of the current work imply that the abundant forest biomass residue of bark could be transformed into high added-value solid biofuels, especially through the examination of each species' properties and potential individually, as well as through the optimization of the wood-to-bark ratio of solid biofuel feedstocks. In this way, new opportunities would undoubtedly open for local industries and alternative residual materials would contribute to the saving of the valuable wood raw material.

4. Conclusions

The main conclusions that could be drawn from the current work are summarized in the following:

- The use of black pine bark as a feedstock for the production of wood pellets resulted in smoother and moderately denser pellets, which also demonstrated higher mechanical strength than those made of pure wood. In fact, the increased bark ratio resulted in further improvement of the above properties. Bark presence in pellet feedstock could provide the opportunity for pellets to satisfy properties to be produced at lower temperatures than those of pure wood, reducing the energy consumption and production cost;
- The optimal densification temperature was 120 °C while lower or higher temperatures resulted in inferior properties of the produced properties;
- It can also be concluded that black pine bark could be considered as a raw material or an additive for the production of solid biofuels or other densified materials, as long as the other important properties fulfill the corresponding technical requirements for each application;
- The surface roughness was weakly correlated to the radial compression strength and density of the produced pellets;
- Low black pine bark contents, of up to 11.3%, 20%, and 36%, could ensure the production of fuel pellets that comply with quality requirements of A1, A2, and B classes, respectively, all of which correspond to residential use.

Author Contributions: Conceptualization, C.L. and V.K.; methodology, C.L. and V.K.; software, C.L.; validation, C.L. and V.K.; formal analysis, C.L., V.K. and G.I.M.; investigation, V.K.; resources, V.K.; data curation, C.L. and V.K.; writing—original draft preparation, V.K.; writing—review and editing, C.L. and G.I.M.; visualization, C.L.; supervision, C.L. and V.K.; funding acquisition, C.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research work was funded by the University Forest Administration and Management Fund of Aristotle University of Thessaloniki (Thessaloniki, Greece) in the context of “Small Research Projects” funding and competition.

Data Availability Statement: Research data of the present manuscript are available upon request to the corresponding author.

Acknowledgments: The authors would like to thank Paschalina Terzopoulou, and Ioannis Barboutis, a former faculty member, of AUTH for their valuable assistance.

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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