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Valorization of Cork Residues for Biomass Pellet Production: Meeting ENplus® Standards Through Strategic Blending

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Abstract: Cork processing generates significant by-products that pose environmental challenges and waste management concerns. This study investigates the potential of utilizing cork residues—finishing powders, grinding powders, and sawdust—for biomass pellet production, emphasizing compliance with ENplus® A1, A2, and B standards. Physical, chemical, and calorimetric analyses reveal that sawdust is the only material capable of independently meeting ENplus® requirements, due to its low nitrogen (0.19%) and ash (0.22%) contents. However, its low net heating value necessitates blending with cork residues for improved energy performance. Finishing powders, despite a high net heating value (17.36 MJ/kg) and low ash content (0.37%), are restricted by their elevated nitrogen levels (1.59%). Grinding powders, with net heating values ranging from 16.25 to 17.78 MJ/kg, offer limited suitability due to high ash and nitrogen contents. For Class A1, mixtures require 85–87% sawdust, limiting cork residue incorporation to 15%. For Class A2, sawdust inclusion drops to 65–70%, allowing for greater use of cork residues and boosting net heating values to 16.74 MJ/kg. Class B mixtures achieve the highest incorporation of cork residues (up to 65%), with net heating values reaching 16.92 MJ/kg, suitable for industrial applications. These results highlight blending strategies as essential for balancing regulatory compliance, energy efficiency, and waste valorization. Future research should focus on pretreatment methods, alternative biomass sources, and lifecycle assessments to enhance compliance and scalability, promoting sustainable energy solutions and circular economy goals.

Keywords: cork residues; biomass pellets; ENplus® standards; sustainable energy; circular economy; waste valorization

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1. Introduction

Industrial waste management in sectors like cork processing poses environmental challenges, with 25–30% of raw material in Portugal's cork stopper manufacturing becoming waste [1]. These by-products, including finishing powders, sawdust, and grinding residues, exhibit complex chemical and physical characteristics, which complicate their management [2]. Improper handling of these residues can lead to soil, water, and air contamination, while simultaneously representing a loss of potentially valuable resources.

These residues contain phenolic compounds, tannins, and suberin, which are resistant to natural degradation and can be toxic to aquatic and terrestrial ecosystems [2–7]. Tannins can disrupt soil health and biodiversity by inhibiting plant growth and microorganism

activity, altering its chemical and physical properties, and compromising fertility and plant life support [3].

Suberin, a hydrophobic biopolymer present in cork, contributes to the resistance of these residues to biological degradation [2,4], hindering its natural decomposition, increasing their environmental accumulation and potential long-term negative impacts [4,5,8]. The improper management of solid cork industry residues, such as their disposal in landfills, can result in the leaching of these compounds into soil and groundwater, causing contamination and affecting water quality [3,4].

Uncontrolled burning of cork residues releases pollutants, affecting air quality and public health [9]. Implementing waste management strategies and converting cork residues into biomass pellets can mitigate environmental impacts and promote circular economy and sustainability [10–12].

Several scientific studies support the viability of this approach, demonstrating the potential of cork residues for energy production in various technological contexts. For example, the Enercork project [13] assessed the implementation of an energy conversion system to maximize thermal energy production from cork dust, with partial utilization for cogeneration of electricity. Additionally, Amorim Cork S.A. installed a combined heat and power (CHP) system in one of its white cork agglomerate production units for coatings [10].

Ref. [14] investigated the anaerobic digestion of cork boiling wastewater (CBW). Their results showed that CBW, despite its challenging composition, could produce methane with significant energetic potential. Ref. [15] explored the treatment and energy valorization of cork industry effluents through anaerobic digestion and co-digestion with food waste and cattle manure, showing that co-digestion improves biogas production, suggesting an efficient solution for treating these effluents and extracting energy value.

Ref. [10] emphasized the widespread use of cork powder in biomass boilers for industrial heat production, highlighting its high thermal efficiency in combustion systems. Similarly, Ref. [16] found that cork residues, including sawdust, sandpaper dust, and triturated wood, can be gasified to produce combustible gases for electric power generation, highlighting the thermochemical potential of cork waste for renewable energy applications. Ref. [17] explored the transformation of cork residues into various bioenergy forms, focusing on macroscopic energy systems, highlighting cork's versatility as a renewable energy source.

These studies highlight that cork industry residues can be used as renewable energy sources, promoting environmental sustainability and energy efficiency. However, challenges in producing high-quality biomass pellets from these residues remain unresolved. To make these solutions commercially viable and environmentally responsible, it is crucial to ensure they meet stringent quality and safety standards like ENplus® A1 and ISO 17225-2. The unique chemical composition of cork residues, particularly their high suberin and nitrogen content [18], presents significant barriers to achieving compliance with rigorous standards like ENplus® A1 and ISO 17225-2 [19,20]. The ENplus® A1 standard and ISO 17225-2 are crucial standards in the industry for pellets. They outline parameters like ash content, nitrogen levels, and pellet stability, ensuring efficient performance in modern boilers and stoves, and provide a technical framework for classifying solid biofuels, ensuring compatibility with combustion technologies and minimizing environmental impacts [19,20]. Non-compliance with pellet standards can hinder market acceptance and limit their use in residential heating systems. Technical issues like nitrogen oxide emissions, ash formation, and pellet instability can also affect energy efficiency and environmental sustainability [21]. The production of compliant pellets for cork residues is crucial as it limits their industrial application, reduces their marketability, and restricts their potential to replace conventional biomass sources. This requires practical adjustments,

such as blending different types of biomasses to achieve desired chemical composition and regulatory compliance. This not only ensures product viability but also promotes process sustainability [22]. Moreover, addressing this gap is essential for the cork industry to align with broader sustainability goals, including the circular economy and carbon-neutral energy production [23].

The conversion of cork industry residues into high-quality biomass pellets offers a unique chance to tackle environmental issues and promote circular economy principles. The valorization of biomass residues is not only an environmental imperative but also a strategic opportunity aligned with the European Union's Renewable Energy Directive (RED II and RED III) [24,25]. These directives promote the use of sustainable bioenergy sources and prioritize the exploitation of residues and waste materials, in line with the waste hierarchy and circular economy principles. Cork-based residues, if properly characterized and managed, may qualify as advanced biofuels or renewable fuels of non-biological origin, thereby contributing to EU decarbonization targets and benefiting from incentive schemes such as green certification or biomass sustainability criteria.

This approach improves resource efficiency, extends raw material lifecycle, and repurposes cork residues as energy, adhering to stringent standards like ENplus® A1 and ISO 17225-2, promoting responsible resource use and sustainable industrial practices. This study embodies the essence of a closed-loop system, transforming waste into resource, reducing the cork industry's environmental impact and promoting sustainable energy systems. Specifically, this study addresses a gap in the literature regarding the valorization of finishing and grinding cork powders through targeted blending strategies designed to meet ENplus® A1, A2, and B certification standards.

2. Materials and Methods

This study focuses on five distinct types of residues—finishing powder; grinding powders A, B, C, and D; and sawdust—provided by Amorim Cork S.A., Santa Maria de Lamas, Portugal. Each residue type was subjected to a series of tests to determine its physical, chemical, and calorimetric properties. The methodology follows standardized protocols to ensure accurate and reproducible results, forming the foundation for designing pellet mixtures that comply with ENplus® certification standards.

2.1. Sampling

Five types of biomass residues derived from cork processing were analyzed (Table 1). These residues were supplied by Amorim Cork S.A. and categorized as follows:

1. Finishing powder;
2. Grinding powders A, B, C, and D;
3. Sawdust.

Table 1. Description of materials.

Material	Origin	Composition	Key Characteristics	Utility
Finishing Powder	Produced during mechanical finishing operations (e.g., rectification, capping, chamfering)	Natural cork stoppers: Entirely cork Agglomerated cork stoppers: Cork and polyurethane glue	Composed of the mixture of natural and agglomerated cork stoppers residues Particles are fine and uniform	Used for further processing or as filler material
Grinding Powder A	Result of grinding operations (rejected material, shavings, and stopper production waste)	Cork powder, dirt, and some wood	High silica content Low particle size	Unsuitable for cork-based products

Table 1. *Cont.*

Material	Origin	Composition	Key Characteristics	Utility
Grinding Powder B	Similar to powder A but sourced in a different factory	Cork powder, dirt, and some wood	Higher silica and wood content compared to powder A	Unsuitable for cork-based products
Grinding Powder C	Similar to powders A and B but with higher cork content	Cork powder, dirt, and some wood	Higher cork fraction and lower silica content compared to powders A and B	Unsuitable for cork-based products
Grinding Powder D	Result of further processing of stoppers	Cork and wood	High cork and wood content	Unsuitable for cork-based products
Sawdust	Produced during wood operations for capsule manufacturing	Predominantly wood	Fine, uniform particles	Used in energy production

2.2. Density Measurement

The density of each biomass sample was measured using a fixed volume of approximately 1 cm^3. To minimize the impact of voids, present in the biomass particles, the measurement process was repeated four times for each sample. The final density value for each sample was calculated as the mean of these measurements, and uncertainties were determined based on the standard deviation across the replicates.

2.3. Proximate Analysis

A proximate analysis, which included measurements of moisture, volatile matter, ash, and fixed carbon, was performed in duplicate for all biomasses, following ISO 18134-3 [26], ISO 18122 [27], and ISO 18123 [28]. Representative portions of each sample were selected, and the initial mass of each portion was recorded. Moisture content was determined by placing the samples in a Protherm PLF 100/6 furnace (Protherm, Ankara, Turkey) at 105 °C until their weight stabilized. The difference between the initial and final masses was used to calculate the moisture percentage, expressed on a wet basis.

A portion of the dried sample from the moisture analysis was weighed and heated in a sealed crucible at 550 °C until no further weight loss was observed. This procedure allowed for the determination of volatile matter content as the weight difference represented the organic compounds volatilized during heating. The remaining residue was subjected to further heating at 550 °C in a muffle furnace until a constant weight was achieved, representing the ash content. Fixed carbon content was calculated indirectly by subtracting the moisture, volatile matter, and ash percentages from 100%.

To ensure the accuracy of the furnace during all procedures, Type K thermocouples were used for calibration. The calibration process involved verifying the furnace's temperature stability and precision against reference values, ensuring consistent performance throughout the range of operating temperatures.

2.4. Elemental Analysis

Elemental composition analysis was conducted for all biomasses, to determine the concentrations of carbon (C), hydrogen (H), nitrogen (N), and sulfur (S), following the ISO 16948 standard [29]. The oxygen (O) content was calculated as the remainder after subtracting the percentages of C, H, N, S, and ash from 100%. All analyses were performed in duplicate using a ThermoScience FlashSmart elemental analyzer (Waltham, MA, USA), which was calibrated for accuracy and precision using certified standards, including sulfanilamide, cystine, and BBOT (1,3-butanediol bis(4-benzyloxycarbonyl)oxyethyl ester).

2.5. Calorimetry

The net heating value (NHV) of all biomasses was determined using an isoperibolic calorimeter (Model 6300, Parr Instruments Co., Moline, IL, USA), following the ISO 18125 standards [30]. Approximately 1 g of each sample was combusted under controlled conditions, and each analysis was performed in duplicate to ensure reproducibility. The calorimeter was calibrated prior to analysis using a certified benzoic acid standard (Parr Benzoic Acid No. 3415, Moline, IL, USA) to ensure the accuracy of the measurements. NHV calculations adhered strictly to the procedures outlined in ISO 18125.

2.6. Mixture Design and Rationale

The mixtures were developed based on the availability of residues provided by Amorim Cork S.A. and their physical and chemical properties characterized in this study. Due to confidentiality constraints, the exact quantities of each residue type cannot be disclosed. However, the mixture proportions were optimized to maximize resource utilization, deplete the available stock, and ensure compliance with ENplus® pellet standards. By balancing the different residue types, the mixtures were designed to achieve high pellet quality and align with principles of sustainable waste management.

The ENplus® certification classes follow the International Standard ISO 17225-2, with stricter quality specifications for Class A1, which is intended for use in residential boilers and stoves, and progressively relaxed thresholds for Classes A2 and B, suitable for larger installations or industrial applications. The relevant specifications are summarized in Table 2.

Table 2. ENplus® classification specifications for pellet properties (Source: data from Ref. [19], adapted by the authors).

Property	Unit	ENplus® A1	ENplus® A2	ENplus® B
Moisture	(%)		≤10	
Ash	(%)	≤0.7	≤1.2	≤2.0
Nitrogen	(%)	≤0.3	≤0.5	≤1.0
NHV	MJ/kg		≥16.5	
Sulphur	(%)	≤0.04		≤0.05

To determine the thermochemical properties of a given mixture, a weighted average was calculated. This was carried out by summing the product of each material's percentage in the mixture and its corresponding property value. Although this methodology provides a practical estimation of mixture properties, it does not account for potential synergistic or antagonistic effects during pelletization or combustion. Future work will include experimental validation of the mechanical and combustion performance of the blended pellets. Using this approach, mixtures were optimized to maximize the utilization of available residues, deplete existing stock, and produce pellets that meet the ENplus® A1 classification whenever possible. Class A1 pellets are premium quality, generating minimal ash and meeting the strictest requirements for private household use. Mixtures that could not meet A1 specifications were tailored to meet the less stringent thresholds for Classes A2 or B, which are suited for larger-scale or industrial applications.

It should be noted that ENplus® certification encompasses a broader range of standards across the entire production chain, including raw material selection, pellet quality, and packaging. In this study, only the pellet quality specifications listed above were used for comparison.

3. Results and Discussion

3.1. Characterization of Biomass Samples

The density of the biomass samples was measured to evaluate and assess their potential for further processing, such as densification for pellet production. Measurements were carried out using a fixed volume of approximately 1 cm^3, and the process was repeated four times for each sample to minimize the influence of voids present in the biomass particles. The density results of the biomass samples (Table 3) demonstrate significant variability among the different types of residues, reflecting their diverse compositions and physical structures. The measured densities range from 75.30 kg/m^3 for finishing powder to 391.98 kg/m^3 for grinding powder C, highlighting the differences in material compactness and particle composition.

Table 3. Biomass samples density.

Biomass Sample	Density (kg/m^3)
Finishing powder	75.30 ± 1.03
Grinding powder A	378.06 ± 7.79
Grinding powder B	321.32 ± 9.40
Grinding powder C	391.98 ± 7.22
Grinding powder D	278.17 ± 10.18
Sawdust	223.50 ± 6.87

Finishing powder exhibited the lowest density at 75.30 kg/m^3, which aligns with its fine, lightweight nature and the presence of air spaces within its loose particles [31]. These voids, commonly observed in irregular and loosely packed biomass materials, limit the packing efficiency and contribute to the material's poor agglomeration properties [31]. This low density poses challenges for achieving uniform mixtures and highlights the material's poor agglomeration properties. When compared to sawdust, a material with a density of 223.50 kg/m^3 often used as a binder, it becomes clear that incorporating finishing powder into pellet production will require special care to ensure a uniform and cohesive pellet mixture.

Grinding powders A, B, and C exhibited the highest densities among the samples, consistent with expectations based on their descriptions. Grinding powder C presented the highest density at 391.98 kg/m^3, followed by A at 378.06 kg/m^3 and B at 321.32 kg/m^3. These densities, being relatively closer to sawdust, suggest that these materials are more compatible for forming uniform mixtures. Grinding powder D, on the other hand, demonstrated a lower density of 278.17 kg/m^3, which, combined with its higher particle size and wood content, introduces greater variability and results in a higher measurement uncertainty. The air pockets that form around its larger particles may affect mixture uniformity [31], but given its density is comparable to sawdust, it should not present significant difficulties in producing a homogeneous mixture.

Overall, the density results suggest that most materials, particularly the grinding powders and sawdust, are suitable for achieving uniform mixtures. However, the incorporation of finishing powder requires special attention due to its significantly lower density, which could impact both the uniformity of mixtures and the structural integrity of the resulting pellets. These findings underscore the need to optimize mixture compositions by balancing low- and high-density materials to ensure compliance with quality standards and produce pellets with desirable structural and thermal properties.

Comparing these results to intact cork specimens reported by Ref. [32], we observe significant differences due to the structural and compositional changes introduced during industrial processing. The study by Ref. [32] categorized cork, with mean densities for each

class ranging from 139 kg/m^3 to 211 kg/m^3 depending on the trial direction (radial, axial, or tangential).

Grinding powders A, B, and C (378.06 to 391.98 kg/m^3) far exceed the highest density class reported for intact cork (250 kg/m^3) [32]. This substantial difference reflects the incorporation of non-cork components, such as wood and silica, during processing, which increases density. Grinding powder D (278.17 kg/m^3) is slightly above the upper limit of the cork density range but lower than grinding powders A, B, and C. On the other hand, finishing powder (75.30 kg/m^3) and sawdust (223.50 kg/m^3) align more closely with the natural porosity and lightweight nature of cork [32].

The density results offer valuable insights into the physical characteristics of the biomass samples and their implications for mixture uniformity and pellet quality. However, density alone is insufficient to determine the overall suitability of these materials for pellet production. To complement this understanding, the proximate analysis examines key chemical properties like moisture, volatile matter, ash, and fixed carbon content, providing a comprehensive evaluation of materials' performance and ENplus® standards compatibility, assessing how each biomass type's intrinsic properties influence combustion behavior and compliance.

Interestingly, the finishing powder exhibited the lowest moisture content among all samples, at just 3.23% (Table 4). This low moisture level partially explains its poor agglomeration performance, as both studies and pellet mill manufacturers consistently recommend an ideal moisture content of around 15% for effective pelletizing [1]. The reduced moisture in finishing powder is expected, given its origin from materials subjected to extensive heating and drying processes during cork expansion and sterilization [1].

Table 4. Proximate analysis (dried basis).

Biomass Sample	Moisture (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)
Finishing powder	3.23 ± 0.15	78.85 ± 0.13	0.37 ± 0.06	17.55 ± 0.09
Grinding powder A	9.45 ± 0.30	62.61 ± 0.17	5.03 ± 0.42	22.91 ± 0.21
Grinding powder B	8.36 ± 0.16	62.50 ± 0.63	5.00 ± 0.07	24.14 ± 0.46
Grinding powder C	9.22 ± 0.28	61.43 ± 0.30	3.83 ± 0.10	25.52 ± 0.34
Grinding powder D	8.63 ± 0.30	64.50 ± 0.40	2.22 ± 0.07	24.65 ± 0.54
Sawdust	8.20 ± 0.25	74.14 ± 0.38	0.22 ± 0.001	17.45 ± 0.45

Grinding powders A, B, and C exhibited moisture levels ranging from 8.36% to 9.22%, aligning closely with the values typically observed for conventional biomass, such as the 8.20% recorded for sawdust. Similarly, grinding powder D showed a slightly higher but comparable moisture content of 8.63%. These findings are consistent with the published literature on cork residues, which reports moisture content values typically falling within the range of 8% to 9% [22,33,34]. In general, all biomass samples will require additional moisture to reach the optimal 15% level recommended for pellet production, with special attention needed for finishing powders due to their particularly low moisture content [1].

With respect to volatile matter, grinding powders exhibit the lowest values among the samples, indicating they are likely to be more resistant to combustion [35]. Finishing powder exhibited the highest volatile matter content at 78.85%, followed by sawdust at 74.14%. Consequently, these two samples also had the lowest fixed carbon content, approximately 17%, while grinding powders displayed higher fixed carbon levels around 25%. Higher volatile matter in cork is generally associated with easier combustion due to lower temperatures' oxidation of volatile components [35]. However, despite its high volatile matter content, cork exhibits exceptionally low thermal conductivity and contains

suberin, a compound that decomposes at elevated temperatures, releasing gases that actively suppress combustion [4,36].

Regarding ash content, sawdust recorded the lowest value at 0.22%, followed closely by finishing powder at 0.37%. These figures align with the ash content typically observed in commercial pellets, highlighting their suitability for high-quality pellet production. In contrast, the grinding powders exhibited significantly higher ash content, ranging from 2.22% for grinding powder D to approximately 4.62% for the other grinding powders. This value corresponds to the average ash content of powders A, B, and C, which are later considered as a single material due to their similar characteristics. These elevated values are consistent with findings from other studies on cork waste, which reported ash contents between 4% and 5% for mixtures derived from similar types of residues [22,33,34]. The consistently high ash content observed in cork waste poses a significant challenge, as it implies shorter boiler cleaning and maintenance intervals. To mitigate this issue, blending these materials with biomass that has a lower ash content is recommended, effectively diluting the overall ash level in the resulting mixture. In the case of finishing powder, the recorded ash content of 0.37% is notably lower compared to the 2.19% reported for granules derived from cork stopper finishing operations in the literature [34] or for finishing powders of the same operation with 0.9% [1]. This discrepancy may reflect differences in material handling, processing methods, or regional variations in the raw materials.

The lower volatile matter and higher ash content observed in grinding powders can be attributed to their high silica content, as noted in the material description. This silica-rich composition presents challenges, such as the formation of silicates during combustion, which can lead to ash deposition, reduced heat transfer, and potential mechanical failure or corrosion of combustion equipment at moderate and high temperatures [37]. These characteristics suggest a less efficient combustion process and increased maintenance requirements for boilers. Furthermore, the high ash content in grinding powders aligns with their relatively lower carbon content observed in the elemental analysis (Table 5). Conversely, the elevated volatile matter in finishing powder, which typically facilitates easier combustion, corresponds to its higher hydrogen content. However, this characteristic may decrease the net heating value due to increased water vapor production during combustion.

Table 5. Elemental analysis of the biomass samples (dried basis).

Biomass Sample	N (%)	C (%)	H (%)	S (%)	O (%)
Finishing powder	1.59 ± 0.27	68.04 ± 6.40	8.61 ± 0.86	n.d.	21.40 ± 7.40
Grinding powder A	0.95 ± 0.08	50.22 ± 1.80	4.73 ± 0.23	n.d.	39.07 ± 2.02
Grinding powder B	1.01 ± 0.05	52.65 ± 3.25	5.05 ± 0.38	n.d.	36.29 ± 3.67
Grinding powder C	0.59 ± 0.03	51.33 ± 2.45	4.99 ± 0.41	n.d.	39.26 ± 2.82
Grinding powder D	0.63 ± 0.07	51.98 ± 1.53	5.49 ± 0.46	n.d.	39.68 ± 1.91
Sawdust	0.19 ± 0.10	48.16 ± 1.00	4.98 ± 0.02	n.d.	46.46 ± 0.87

n.d.—not detected.

The elemental analysis reveals elevated nitrogen levels across all samples, with the exception of sawdust, which has a comparatively low nitrogen content of 0.19%. Nitrogen, a natural component of combustion air alongside oxygen, reacts at high combustion temperatures to form nitrogen oxides (NO_x gases). In the lower temperature range typical of biomass combustion, however, it is predominantly the nitrogen within the biomass itself that reacts to produce these compounds [38]. While combustion testing was not conducted in this study, the elevated nitrogen levels observed, particularly in finishing powder, suggest a potential risk for increased NO_x emissions. Nitrogen content is particularly significant in the context of pellet certification standards such as ENplus®, which set a maximum allowable nitrogen level of 1% for Class ENplus® B. This creates a challenge for

the use of finishing powder, which has a nitrogen content of 1.59%, significantly exceeding this threshold.

Moreover, this value is substantially higher than the 0.35% nitrogen content reported for granulated cork in other studies [34]. In contrast, sawdust exhibits a nitrogen content of 0.19%, closely matching other literature-reported values, such as 0.13% [39], reinforcing its suitability for meeting certification requirements and typical nitrogen levels in woody biomass, which average around 0.16% [40]. For grinding powders C and D, similar results were found ranging from 0.33% to 0.57% in cork residues, and even though A and B are slightly higher around 1%, this result is comparable to and even lower than for other forest biomass residues [39].

Regarding carbon content, the analysis reveals that most samples have values around 50%, aligning with the typical range for biomasses, which generally varies from 35% to 65%, with 50% often considered a default benchmark [41]. However, the finishing powder stands out with a significantly higher carbon content of 68.04%, likely reflecting its origin from refined cork materials. Notably, no trace of sulfur was detected in any of the samples, a positive finding for combustion applications as sulfur can lead to the formation of harmful emissions and equipment corrosion.

Oxygen content was highest in sawdust at 46.46%, followed by the grinding powders, which exhibited similar values ranging from 36.29% to 39.68%. These findings align with trends observed in the literature, where the distribution of hydrogen and oxygen content in biomass samples often shows a strong correlation [42]. The finishing powder sample recorded the lowest oxygen content at 21.40%, further emphasizing its distinct thermochemical characteristics. Higher oxygen content generally reduces the amount of external air required for combustion, thereby decreasing the energy input needed to sustain the reaction [43]. Moreover, a low elemental oxygen-to-carbon (O/C) ratio has been associated with increased fuel energy content, complementing the influence of the hydrogen-to-carbon (H/C) ratio in determining the overall energy potential of biomass [44].

With regard to hydrogen content, the finishing powder sample displayed an exceptionally high value of 8.61%, distinguishing it from the other samples. This elevated hydrogen content directly influences the net heating value (NHV) by increasing the production of water vapor during combustion, which contributes to a greater discrepancy between the gross heating value (GHV) and NHV for this material. In contrast, the hydrogen content in all other samples was approximately 5%, about 1% lower than values typically reported for sawdust and other woody biomass in the literature [39]. Similarly, the hydrogen content in the grinding powders was also lower when compared to other cork biomass residues [33], emphasizing the distinct thermochemical characteristics of these materials. The high hydrogen content in finishing powder, despite its potential to decrease the NHV due to increased water vapor production, aligns with its elevated carbon content to suggest significant energy release potential. Similarly, the relatively balanced carbon and hydrogen levels in the grinding powders indicate their suitability for generating consistent heating values. These elemental traits directly influence the calorimetric results, as reflected in the heating values (Table 6).

The grinding powders, with their relatively consistent NHV values ranging from 16.25 to 17.78 MJ/kg, demonstrated suitability for energy applications, particularly when blended to mitigate ash-related issues. Compared to typical forest biomass, which often has NHV values between 15 and 17 MJ/kg, cork residues hold a competitive advantage, offering higher energy content alongside potential sustainability benefits. Grinding powder D achieved the highest NHV at 17.79 MJ/kg.

Table 6. Heating values of the biomass samples.

Biomass Sample	GHV (MJ/kg)	NHV (MJ/kg)
Finishing powder	19.22 ± 0.41	17.36 ± 0.41
Grinding powder A	17.42 ± 0.07	16.25 ± 0.07
Grinding powder B	18.96 ± 0.19	17.62 ± 0.19
Grinding powder C	18.43 ± 0.01	17.22 ± 0.01
Grinding powder D	19.09 ± 0.62	17.78 ± 0.62
Sawdust	17.65 ± 0.34	16.45 ± 0.34

The finishing powder exhibited the most significant difference between GHV (19.22 MJ/kg) and NHV (17.36 MJ/kg), which can be linked to its elevated nitrogen and hydrogen content. High nitrogen levels contribute to energy loss through NO_x formation, while hydrogen generates water vapor during combustion, further reducing the effective NHV. This aligns with findings in other studies where cork by-products, especially finishing powders, exhibit unique thermochemical properties that impact their calorimetric performance [11,45].

Although the net heating values obtained for the cork residue mixtures are competitive with conventional biomass fuels, it is acknowledged that thermochemically upgraded materials such as torrefied biomass or biochar often exhibit higher calorific values. For instance, cork-derived biochar produced via slow pyrolysis has been reported to reach HHV of up to 32 MJ/kg [46]. However, such materials typically require additional energy input and processing infrastructure, which may reduce their cost-effectiveness in decentralized applications [46].

The comprehensive characterization of cork-derived residues and sawdust underscores their diverse physical and chemical properties, which directly influence their suitability for pellet production. While sawdust stands out as a reliable base material due to its compliance with stringent pellet standards, the unique properties of cork residues, such as the high NHV of finishing powders or the ash-rich composition of grinding powders, highlight their potential as supplementary components in pellet mixtures. However, their limitations, including high nitrogen and ash contents, demand careful consideration in mixture formulation to ensure compliance with quality standards. These findings set the stage for exploring how strategic blending of these materials can optimize pellet performance, as detailed in the subsequent section on pellet mixture performance.

3.2. Performance of Pellet Mixtures

Building on the individual characterization results, the following analysis focuses on how specific combinations influence compliance with ENplus® standards.

The usability of cork residues for pellet production, as evaluated through their physical, chemical, and calorimetric properties, highlights significant challenges and opportunities. Finishing powders, with the lowest density of 75.30 kg/m^3, are unsuitable as a base material due to the difficulty of achieving uniform mixing with other materials. Additionally, their low moisture content necessitates water addition prior to pelletizing. However, finishing powders exhibit certain advantageous characteristics, such as a high net heating value (NHV) compared to sawdust and a lower ash content relative to grinding powders, making them more suitable as an additive to enhance specific properties of the final pellet. The main drawback of finishing powders is their elevated nitrogen content (1.59%), which could restrict their incorporation due to the stringent ENplus® standards.

Similarly, the grinding powders exhibit limitations. Grinding powder D, despite having the highest NHV (17.79 MJ/kg), shows high ash and nitrogen contents, making it unsuitable as a base material. Grinding powders A, B, and C are chemically and physically

similar due to their shared origin in the cork manufacturing process, but slight variations, such as higher cork content in powder C or differing processing locations for powder B, lead to minor differences. As individual components, these grinding powders do not present significant advantages. However, combining these materials can homogenize nitrogen and ash levels, making them more viable for meeting ENplus® pellet quality standards.

In contrast, sawdust emerges as the most promising base material, being the only sample to meet all ENplus® requirements, including nitrogen levels. Its low ash and nitrogen contents, along with its compatibility with other materials, make it a strong candidate for blending with cork residues to produce high-quality pellets.

Given their similar physical and chemical properties and shared origin in the same manufacturing processes, grinding powders A, B, and C are treated as a single material for the purpose of calculations. This combined material, referred to as 'Grinding powder A, B, C', is characterized by averaging the properties of the individual powders. This approach ensures a representative depiction of their performance in mixture formulations, while simplifying the analysis. It is also important to note that the chemical composition of cork residues can vary considerably depending on the industrial origin and processing method. A previous study using cork powder from a different supplier reported significantly different nitrogen and ash contents [47], underscoring the inherent heterogeneity of cork-based materials and the need for source-specific characterization.

Class ENplus® A1 pellets have stringent limits on ash and nitrogen content, set at 1% and 0.3%, respectively. Among all the materials analyzed, only sawdust meets both of these requirements, as shown in Tables 4 and 5. Consequently, sawdust was incorporated as the primary component, comprising 85% to 87% of the three proposed mixtures (Table 7).

Table 7. Class ENplus® A1 pellet mixtures.

	Mixture 1	Mixture 2	Mixture 3
Composition (%):			
Finishing powder	2.00	0.00	5.00
Grinding powder (A, B, C)	8.00	7.00	2.00
Grinding powder D	5.00	7.00	6.00
Sawdust	85.00	86.00	87.00
Properties:			
Moisture (%)	8.190 ± 0.213	8.290 ± 0.213	7.990 ± 0.213
Ash (%)	0.670 ± 0.057	0.670 ± 0.057	0.430 ± 0.057
Nitrogen (%)	0.290 ± 0.084	0.270 ± 0.085	0.300 ± 0.087
NHV (MJ/kg)	16.560 ± 0.291	16.596 ± 0.296	16.596 ± 0.299
Sulphur (%)	N.D.	N.D.	N.D.

N.D.—not detected.

Finishing powder, despite having an ash content of less than 1%, cannot be incorporated in large quantities due to its exceptionally high nitrogen content of 1.59%. This limitation is evident in mixture 3, where just 5% inclusion of finishing powder is enough to reach the nitrogen limit of 0.30%. Sawdust, while exhibiting low nitrogen and ash content, has the lowest NHV among the samples, followed by the grinding powders. Consequently, mixture 1 barely meets the NHV requirement of 16.560 MJ/kg for Class ENplus® A1 pellets. Mixtures 2 and 3 achieve higher NHVs of 16.596 MJ/kg, attributed to the inclusion of 7% grinding powder D in mixture 2, which has the highest NHV among the samples, and a combination of grinding powder D (6%) and finishing powder (5%) in mixture 3, which also contributes its relatively high NHV. Overall, the availability of materials that can fully meet all Class ENplus® A1 requirements is significantly limited, with sawdust being the only biomass that fully complies.

To address the challenges of meeting Class ENplus® A1 standards, formulations were adjusted to align with the more lenient requirements of Class ENplus® A2. In these mixtures, nitrogen content emerged as the critical limiting factor, with Class ENplus® A2 allowing for a maximum of 0.5%. Among all the materials analyzed, sawdust remained the only biomass capable of ensuring compliance, necessitating its significant inclusion in the mixtures. This resulted in sawdust comprising 65% of mixture 1 and 70% of mixtures 2 and 3 (Table 8). While ash content neared the acceptable threshold in mixture 1, owing to its lower sawdust concentration and higher inclusion of grinding powders, including grinding powder D, the determining factor across all three mixtures was the nitrogen content, which remained close to the limit at 0.49% in mixtures 1 and 2, and slightly lower at 0.47% in mixture 3.

Table 8. Class ENplus® A2 pellet mixtures.

	Mixture 4	Mixture 5	Mixture 6
Composition (%):			
Finishing powder	11.00	15.00	11.00
Grinding powder (A, B, C)	18.00	12.00	19.00
Grinding powder D	6.00	3.00	0.00
Sawdust	65.00	70.00	70.00
Properties:			
Moisture (%)	7.830 ± 0.213	7.560 ± 0.213	7.810 ± 0.213
Ash (%)	1.150 ± 0.057	0.830 ± 0.057	1.070 ± 0.057
Nitrogen (%)	0.490 ± 0.071	0.490 ± 0.08	0.470 ± 0.076
NHV (kWh/kg)	16.740 ± 0.233	16.704 ± 0.249	16.668 ± 0.247
Sulphur (%)	N.D.	N.D.	N.D.

N.D.—not detected.

The higher limits allowed under Class ENplus® A2, compared to A1, enable greater inclusion of cork by-products in the mixtures, reducing the reliance on sawdust and consequently increasing the NHV. Among the mixtures, mixture 4 achieved the highest NHV at 16.740 MJ/kg, with a sawdust content of 65%. This was followed by mixtures 5 and 6, both containing 70% sawdust, with NHVs of 16.704 MJ/kg and 16.668 MJ/kg, respectively. The elevated NHV in mixture 4 can be attributed to its lower sawdust content and the highest concentration of grinding powder D (6%). In mixture 5, the NHV decreased slightly due to the increase in sawdust to 70% and the reduction in grinding powder D to 3%.

The further reduction in NHV from mixture 5 to mixture 6 resulted from the complete elimination of grinding powder D and a decrease in finishing powder, replaced by additional grinding powder. Overall, these adjustments demonstrate the feasibility of incorporating higher proportions of cork by-products into the mixtures under Class ENplus® A2 standards.

The shift to Class ENplus® B offers increased flexibility in material composition due to its higher allowable thresholds for nitrogen and ash content. Unlike Classes A1 and A2, where stringent limits necessitated a dominant proportion of sawdust, the Class B requirements enable a broader incorporation of cork by-products, despite the limitation of the usage of these pellets to industrial applications. However, the primary constraint for Class B mixtures becomes the ash content, capped at 2%. Nitrogen remains a factor, with a limit of 1%, though only finishing powder exceeds this threshold. This relaxed framework allows for a significant reduction in sawdust content, with mixtures 7 and 8 containing just 35% sawdust and mixture 9 slightly higher at 40%, enabling greater utilization of the other cork-derived materials (Table 9).

Table 9. Class ENplus® B pellet mixtures.

	Mixture 7	Mixture 8	Mixture 9
Composition (%):			
Finishing powder	25.00	30.00	20.00
Grinding powder (A, B, C)	37.00	35.00	37.00
Grinding powder D	3.00	0.00	3.00
Sawdust	35.00	35.00	40.00
Properties:			
Moisture (%)	7.270 ± 0.213	6.990 ± 0.213	7.520 ± 0.213
Ash (%)	1.940 ± 0.057	1.800 ± 0.057	1.940 ± 0.057
Nitrogen (%)	0.800 ± 0.077	0.840 ± 0.089	0.730 ± 0.068
NHV (kWh/kg)	16.920 ± 0.179	16.920 ± 0.188	16.884 ± 0.181
Sulphur (%)	N.D.	N.D.	N.D.

N.D.—not detected.

Sawdust, with the lowest NHV among the materials, contributes to the highest NHV values in mixtures 7 and 8 at 16.920 MJ/kg, while mixture 9 exhibits a slightly lower NHV of 16.884 MJ/kg due to a 5% increase in sawdust concentration. The high ash content of the grinding powders restricts their concentration to a maximum of 37%, ensuring compliance with the 2% ash limit set by the ENplus® B standard. Similarly, the finishing powder, despite its low ash content of 0.37%, is limited to a concentration of 30% due to its elevated nitrogen content.

Overall, the proposed mixtures for Class ENplus® B facilitate a significant increase in the incorporation of cork by-products, nearly doubling their usage compared to the stricter A1 and A2 Classes. Although combustion testing of the proposed mixtures was not performed in this study, the mixture formulations were designed to meet the chemical requirements of ENplus® standards. Future work will include experimental validation of their combustion performance and emissions.

The ability of optimized mixtures to meet ENplus® standards without pretreatment enhances the practical feasibility of using cork residues for pellet production. Combined with compliance pathways under EU bioenergy frameworks, this may also support their market viability within certified biomass systems [24,25].

4. Conclusions

This study evaluated the potential of cork processing residues for biomass pellet production, focusing on their physical, chemical, and calorimetric properties. Among the individual biomasses, sawdust emerged as the only material capable of meeting the stringent ash and nitrogen content limits required for all ENplus® Classes. Its low nitrogen (0.19%) and ash (0.22%) contents, combined with its compatibility as a base material, make it indispensable for achieving compliance with ENplus® A1, A2, and B standards.

Finishing powder, while offering an impressive NHV (17.36 MJ/kg) and low ash content (0.37%), is limited by its exceptionally high nitrogen content (1.59%), which restricts its inclusion in compliant mixtures, particularly for Classes A1 and A2. Grinding powders A, B, and C share similar properties, including high ash content (4–5%) and nitrogen levels (~1%), making them unsuitable as standalone materials. Grinding powder D, with the highest NHV (17.78 MJ/kg) among the samples, presents slightly lower ash and nitrogen levels than the other grinding powders but remains constrained by these parameters.

For Class ENplus® A1 pellets, sawdust must constitute a dominant 85–87% of the mixture, limiting the incorporation of cork by-products to only 13–15%. This constraint ensures compliance with strict nitrogen and ash limits but results in reduced NHV values, with the highest mixture achieving 16.596 MJ/kg. In contrast, the more relaxed specifications of

Class ENplus® A2 allow for sawdust proportions to drop to 65–70%, enabling the inclusion of additional cork residues. This adjustment raises the NHV of the mixtures to a maximum of 16.740 MJ/kg, showcasing the energy potential of cork-derived materials.

The most notable improvement in cork by-product utilization is seen in Class ENplus® B mixtures, where the permissible ash and nitrogen levels allow for a steep reduction in sawdust content to 35–40%. This flexibility facilitates the incorporation of up to 65% cork residues, nearly doubling their usage compared to A1 mixtures. The highest NHV recorded was 16.920 MJ/kg, attributed to the lower reliance on sawdust and the increased inclusion of high-energy materials such as finishing and grinding powders. However, this limits the utilization of these pellets on industrial applications.

From a sustainability perspective, the findings emphasize the critical role of blending strategies in maximizing the valorization of cork residues while meeting ENplus® standards. Incorporating up to 65% cork by-products in Class B mixtures demonstrates significant progress in waste minimization, aligning with circular economy principles. Nevertheless, challenges such as high nitrogen and ash content persist, underscoring the need for further research into pretreatment methods, such as torrefaction, and alternative blending materials to enhance compliance and energy performance.

This study illustrates the feasibility of utilizing cork by-products for pellet production, with Class ENplus® B mixtures offering the most promising balance between energy density and waste valorization. While sawdust remains indispensable for achieving compliance across all ENplus® Classes, the strategic incorporation of cork residues provides a pathway to sustainable energy production and efficient resource utilization.

Future research should explore ways to improve the use of cork residues in biomass pellet production while adhering to strict standards. Pretreatment techniques like torrefaction or chemical treatments could be explored to reduce nitrogen and ash content, improving energy density and combustion properties. Additionally, incorporating alternative biomass sources with lower levels of nitrogen and ash could align further with ENplus® requirements while maintaining high net heating values. Lifecycle assessments (LCAs) are recommended to comprehensively assess the environmental and economic impacts of cork residue valorization, including energy consumption, greenhouse gas emissions, and overall sustainability benefits. Further investigation into ENplus® pellet quality requirements—such as mechanical durability, fines content, bulk density, pellet length, and combustion performance as well as ash melting behavior—will be crucial to validate their practical application in residential and industrial settings. Furthermore, future studies should investigate potential synergistic or antagonistic interactions between cork residues during pelletization and combustion, which may influence mechanical properties, emissions, and energy conversion efficiency. Pilot-scale production trials are essential to optimize cost-efficiency and address logistical challenges associated with commercialization. Moreover, assessing the variability of cork residues across different processing facilities and seasonal conditions will be important to ensure the consistency and reliability of the blending strategy.

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