

Article

Characterization of Cellulose Derived from Invasive Alien Species Plant Waste for Application in the Papermaking Industry: Physic-Mechanical, Optical, and Chemical Property Analysis

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Featured Application: Papermaking industry and technology. Innovative applications of vegetal residues.

Abstract: This study examines the potential of four invasive plant species, both arboreal and herbaceous, within the riparian forest of the Umia River in Galicia, a common ecosystem in northern Spain. These invasive species (*Arundo donax*, *Phytolacca americana*, *Eucalyptus globulus*, and *Tradescantia fluminensis*) were collected and assessed for their suitability as an alternative source of pulp and paper materials for the paper industry to mitigate the environmental impacts associated with conventional cellulose fiber production from harmful monocultures. Cellulosic material from leaves, bark, and/or stems of each of the selected species was isolated from lignin and hemicelluloses through kraft pulping processes. Resulted fibers and pulps were analyzed visually, morphologically, chemically, and mechanically to evaluate their papermaking properties. To compare these properties with those of commercially available pulp, test sheets were concurrently produced using commercial bleached Eucalyptus cellulose. The findings reveal that the employed fibers exhibit promising characteristics for artistic paper production. Regarding the pulp, two refining times were tested in a PFI machine, and the Schopper–Riegler degree was measured. Paper sheets underwent various tests to determine thickness, basis weight, apparent volume, apparent density, permeability, and chemical composition, as well as microscopic optical and morphological properties. The fibers obtained from the waste derived from the removal of invasive exotic species and biodiversity control present a viable and intriguing alternative for decentralized paper production, yielding noteworthy results for the creative sector. This research highlights the potential of harnessing invasive species for sustainable and innovative paper manufacturing practices.

Keywords: paper and pulp manipulation; invasive alien species waste; physic-mechanical properties; paper industry; circular economy; sustainability



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1. Introduction

The fast development of industry after the industrial era and within the political and economic dynamics of contemporary moment are resulting in an over exploitation of natural resources. This behavior is having a strong impact on the balance and processes in nature, depleting raw materials needed to satisfy diverse needs [1].

The global paper industry faces enduring environmental challenges associated with the extensive adoption of monoculture tree plantations, transcending mere industrial

boundaries. The preponderance of monocultures, characterized by uniform stands of a single tree species, is causally linked to an array of ecological predicaments. These agroforestry systems, designed to cater to the insatiable demand for paper and paper-related products, have engendered a spectrum of ecological quandaries, including but not limited to deforestation, diminished biodiversity, soil degradation, and perturbations in local climatic regimes [2–4]. The ceaseless pursuit of escalating paper output has often exacted an exorbitant toll on the very ecosystems that underpin global ecological health and equilibrium.

Preliminary reports from CEPI [5] in 2021 showed that paper and paperboard production is increasing, becoming one of the largest industries, costing huge amounts of raw material for its functioning. Between 2020 and 2021, global paper and board production increased by about 3%. These increasing ciphers were derived in the effort of the paper industry to decarbonize and innovate biomass origin, and, recently, there is an increasing interest in using agricultural residues [1].

Several initiatives, such as zero waste or circular economy dynamics, are now allowing us to start countering pollution by reintroducing waste into production systems. This approach involves the intelligent planning and design of products from the outset, allowing for the proactive selection and prediction of production processes and resource utilization. In this manner, effective waste management is achieved while creating fresh opportunities for business growth [1]. Circular economy systems prioritize resource efficiency, waste reduction, and sustainable practices to create a closed-loop cycle that minimizes negative environmental impacts.

In relation to these kinds of initiatives, vegetal waste from agroforestry management presents a huge opportunity for the papermaking industry. Moreover, waste derived from the extraction and cleaning of areas affected from Invasive Alien Species seems to be a considerable option for sustainable papermaking, promoting biodiversity care. Some studies performed usually discarded parts of the Eucalyptus, such as the bark or leaves, showing significantly lower yield, delignification degree, and strength properties, but having a quicker response to refining [6]. Other studies, performed with IAS residual biomass [7] and non-woody plants, demonstrated important plant property improvements [8]. Moreover, its possible deficiencies, reintroducing waste or residual materials, different tree species, or annual plants in paper production processes, help to reduce wood cost and biodiversity damage [9–13].

Invasive alien species represent a challenge for natural systems' management. Europe and regions across the globe are grappling with the escalating challenges precipitated by Invasive Alien Species (IAS). These non-indigenous organisms, introduced into ecosystems either intentionally or inadvertently through anthropogenic activities [4], have successfully established themselves in non-native territories, frequently resulting in deleterious consequences for indigenous flora and fauna, perturbing established ecosystems, and occasionally precipitating species extinctions. Reports show that there are an estimated over 12,000 Alien Species in Europe, and about 10–15% are developing invasive behavior [14]. As shown in the European Commission Report of 2016, it is estimated that IAS have cost at least 12 billion/year over past 20 years. Management initiatives and big efforts taken to stop their spreading and trying to control them have demonstrated to cost substantial human, natural, and financial resources.

The distribution of costs and benefits arising from Invasive Alien Species tends to exhibit an inherent imbalance. Individuals or entities that stand to gain from introducing IAS into the European Union often lack economic motivations to mitigate the associated risks. Conversely, the expenses linked to IAS-related damage and control efforts typically burden a broader spectrum of stakeholders, encompassing primary producers, public authorities, and society at large. This asymmetry underscores the pressing need for comprehensive policies and strategies to address the multifaceted challenges posed by IAS and ensure a fair and equitable distribution of both responsibilities and benefits across affected sectors [14].

It is for those reasons that it seems coherent to reintroduce waste derived from the extraction of IAS into paper production systems, reduce waste burns, and obtain benefits that could be reused into natural ecosystems' management to restore certain equilibrium. The utilization of invasive species plant biomass within the ambit of the paper industry proffers a prospective avenue for the attainment of environmental sustainability objectives. By reutilizing the biomass of invasive plants in paper production processes, the potential exists to ameliorate deforestation pressures, alleviate the strain on indigenous forests, and catalyze ecological restoration [15].

The objective of this study is, through the conducted experiments, to demonstrate that papers made from the waste of selected invasive exotic species can be used for pulp and paper production. In addition to establishing the suitability of this material for such purposes, we aim to highlight potential applications for the paper industry, which could be further examined and analyzed in future studies, thus discovering new sources of innovation and ecosystem conservation.

2. Materials and Methods

2.1. Selected Species

Studied species were selected for their major presence in the riverbank ecosystems in Umia river in Galicia, Spain. Due its considerable representation in caparison with local flora and management solutions implemented by the city council, waste was produced from scheduled cleaning operations in the area. Selected Invasive alien species were: *Arundo donax* [16–18], *Phytolacca americana* [19,20], *Tradescantia fluminensis* [17,21], and *Eucalyptus globulus* Labill [22].

The four species were considered due their behavior and ecological characteristics as invasive alien species, even though *Eucalyptus* is not officially recognized as such in Spain. The usual dissociation between ecological and economical interest is what, in some situations, derive into the official consideration or not of a species as an IAS. Economical and industrial interest in *Eucalyptus* as one of the major suppliers of raw matter for paper production in Spain derives into management initiatives that are usually delinked within ecological needs of affected areas.

Raw matter from the species was selected in relation to their mechanical and physical characteristics for pulping and papermaking (Table 1). Also, in *Eucalyptus*' case of study, usually discarded parts were used, such as branches, leaves, and bark. In the case of *Phytolacca americana* and *Arundo donax*, test sheets and pulping tests were developed separately for stems and leaves. On the contrary, *Tradescantia fluminensis* was used completely and indistinctly, showing great potential for special and artistic papers.

Table 1. Parts of raw matter used for handsheet testing and pulping and moisture.

Species	Type	Family	Raw Matter	Moisture (%)
<i>Arundo donax</i>	Herbaceous	Poaceae	Leaves	70.12
			Stem	87.79
<i>Tradescantia fluminensis</i>	Herbaceous	Commelinaceae	Complete	91.92
<i>Phytolacca americana</i>	Herbaceous	Phytolaccaceae	Leaves	80.22
			Stem	67.25
<i>Eucalyptus globulus</i> Labill	Arboreal	Myrtaceae	Leaves	78.89
			Bark/Branches	77.54

2.1.1. *Arundo donax*

Colloquially known as common reed, *Arundo donax* (Figure 1) is a perennial herbaceous species from the Poaceae family, characterized by its aquatic habitat and rhizomatous growth pattern [17]. Notably, it has garnered infamy as one of the most warned against

invasive species due to its fast-growing capacities, particularly prevalent in warm-climate regions encompassing Oceania, Africa, and the Americas [23].



Figure 1. *Arundo donax* species. Photographed in the Umia riverbank area in spring 2023.

The native origins of *Arundo donax* are believed to be rooted in Eurasian regions characterized by Mediterranean and subtropical climates. However, the precise provenance of its invasive populations remains enigmatic [24]. It was first documented in Galicia in 1852, introduced for cultivation purposes [17].

Beyond its invasive characteristics, *Arundo donax* boasts notable mechanical attributes, rendering it suitable for applications in the field of carpentry [25]. Moreover, it assumes significance in terms of environmental conservation, serving as a viable source of raw material for bioenergy production and biochemical processes [26]. Research has unveiled the potential of its chemical constituents in diverse sectors, ranging from industrial applications to medical domains, as prospective antimicrobial agents [23] and antioxidants [17,18]. Furthermore, there is evidence to suggest that the chemical compounds derived from *Arundo donax* possess the capacity to ameliorate soil and aquatic ecosystems afflicted by industrial pollution, enhancing their fertility [23].

2.1.2. *Tradescantia fluminensis*

Commonly known as “Men’s love” and “Cat’s ear” this plant (Figure 2) is a perennial herbaceous plant belonging to the Commelinaceae family, indigenous to South America, with a natural distribution range spanning from southeastern Brazil to Argentina. This species was initially documented in Galicia in 1951, when it was identified as an invasive species. Notably, its introduction into new territories was often driven by ornamental purposes, showcasing its allure in horticultural contexts [21].

It has remarkable capacity for rapid propagation, particularly in environments characterized by low light levels and high humidity, such as dense forests and riparian zones. This proclivity has precipitated adverse consequences for the regeneration of indigenous vegetation in affected areas, posing challenges for ecosystem health and biodiversity [17]. Managing its expansion presents intricate challenges due its chemical resistance to herbicides and an innate capacity for swift recovery, rendering control efforts demanding [21].

Prior investigations have identified the Commelinaceae family as potential sources of renewable bioactive compounds [27]. These compounds have exhibited intriguing health-related properties, including anti-inflammatory, antipyretic, diuretic and antioxidant attributes [17,18] suggesting their potential incorporation into modern medical practices [28].



Figure 2. *Tradescantia fluminensis* species. Photographed in the Umia riverbank area in spring 2023.

2.1.3. *Phytolacca americana*

Phytolacca americana (Figure 3), commonly referred to as American pokeweed, is a perennial herbaceous species from the Phytolaccaceae family, renowned for its dyeing characteristics. Native to Eastern North America, this plant colonizes disturbed environments in humid forests, riverbanks, and territorial boundaries. It exhibits adaptability to a broad range of environmental conditions, encompassing both wet, semi-shaded, and sunny habitats, frequently occupying agricultural settings [20].



Figure 3. *Phytolacca americana* species. Photographed in the Umia riverbank area in spring 2023.

The growth potential of *Phytolacca americana* is substantial, with mature individuals attaining heights of up to 3 m. The species features large, ovate leaves, inconspicuous white flowers, and distinctive dark berries known for their remarkable dyeing capabilities. Rooted in a robust system, *Phytolacca*'s underground apparatus comprises a thick taproot and multiple heads. The stem, characterized by a cylindrical and hollow structure, displays fleshy tissue with distinctive purple or red markings, assuming a woody base. The leaves exhibit a bi-tonal coloration, with the upper surface appearing darker.

The geographical distribution of *Phytolacca americana* transcends its native range in North America, encompassing the Americas and Europe since the 17th century, notably within the Mediterranean region. Furthermore, the species has successfully naturalized in diverse regions, including Turkey, Asia, India, China, Taiwan, Japan, Indonesia, Australia, and New Zealand. It ranks prominently among the globally pervasive Invasive Alien Species (IAS) [19]. Facilitated by avian-mediated seed dispersal and its intrinsic adaptability, *Phytolacca*'s invasion extends to a spectrum of natural habitats, including uncultivated vineyards, orchards, cultivated fields, ruderal zones, and urban landscapes.

2.1.4. *Eucalyptus globulus*

Eucalyptus (Figure 4), a genus within the Myrtaceae family, encompasses an extensive array of over 900 species and subspecies of trees and shrubs [29]. Notable for its remarkable resilience to environmental stressors such as fire, drought, and soil acidity, coupled with its rapid growth rate, this genus holds significant ecological and economic importance. Of particular interest are subspecies like *globulus*, *maidenii*, *biscotata*, and *pseudo globulus*, originating primarily from Tasmania and Australia. *Eucalyptus globulus* gained worldwide prominence, especially in Mediterranean, subtropical, and tropical regions, during the period spanning 1800 to 1850 [30]. It is noteworthy that the first introduction of *Eucalyptus* to Europe dates to 1774 at the Kew Botanical Gardens [22]. Early records in Galicia, documented by W. Weber's catalog of exotic flora [31], initially classified *Eucalyptus* as an invasive species upon its introduction for cultivation in 1955. However, recent reclassification in Spain has redefined its status as a non-native species rather than invasive [18].



Figure 4. *Eucalyptus globulus* species. Photographed in the Umia riverbank area in spring 2023.

An extensive study conducted by the Confederation of Spanish Forestry Organizations (COSE) in 2019, titled “*Eucalyptus* in Galicia: Environmental and Socioeconomic Aspects”, revealed that *Eucalyptus* accounts for 20% of Galicia's forest population and represents 60% of the timber designated for forestry harvesting. Notably, *Eucalyptus* bark, rich in chemical constituents, is frequently discarded when wood is processed for construction, paper production, or charcoal manufacturing. These discarded bark residues are often incinerated for energy generation [32]. However, research indicates their potential in various applications, including soil improvement [33], solvent utilization for aqueous contaminant removal [34], bioethanol production, antioxidant compounds [18], and the development of environmentally friendly adhesives utilizing tannins extracted from the bark [35].

In the wood and paper industry, *Eucalyptus* leaves are typically considered byproducts and are commonly repurposed for soil enrichment or energy generation through combustion. Nevertheless, scientific investigations have unveiled the valuable bioactive properties inherent in this biomass, making it a promising candidate for biorefinery processes [36].

2.2. Harvesting and Delignification Kraft Process

The four highly invasive arboreal and herbaceous plants (*Arundo donax*, *Phytolacca americana*, *Eucalyptus globulus*, and *Tradescantia fluminensis*) were harvested between March and May of 2023 in the Umia Riverbank area, in a delimited area of 2 km in the walking path after Ribadumia town (Pontevedra, Spain). Before the treatment in laboratory, samples were separated into its parts, shown in Table 1 (leaves, stem, and bark/branches), and then fragmented to 50–20 mm long pieces. Prepared raw matter was dried at 105 °C until moisture was reduced.

Once raw matter was dried, delignification of biomass samples was conducted according to kraft process. Delignification processes aim to dissociate lignin from cellulose fibers within lignocellulosic biomass by breaking ester and ether bonds present in the natural lignin structure [37]. Due to the lack of proper digester, dry matter was cooked in 5 L of distilled water within 20% of dry mass weight of NaOH added. Other studies [38] suggested that optimized conditions for the delignification process in a forced circulation digester were: 22% of effective alkali charge (NaOH); 30% sulfidity; a liquor-to-wood ratio of 4:1; a cooking temperature of 165 °C; and 90 min of cooking time. In this occasion, the liquid/solid mass ratio was 5:1, as established in other studies [39]. And, the estimated time of cooking was 120 min at 160 °C, depending on raw matter type (leaves, stem of bark). *Arundo donax* and *Phytolacca americana* stems and *Eucalyptus globulus* bark were the samples that needed more reaction time. Once the fibers were in optimal condition, and all the lignin was already eliminated, it was possible to separate them without any mechanical means.

After the kraft process (Figure 5), and once the reaction was finished, the mixture was filtered and rinsed. Back bleaches, the residual liquid obtained after the delignification process, was then stored for posterior neutralization with wood sawdust. By this means, the pH is neutralized, obtaining an organic matter suitable to be used as compost.



Figure 5. Kraft delignification process developed in the school of Forestry Engineering of the University of Vigo, Galicia.

The unbleached delignified pulp was further dried at 105 °C during at least 24 h and divided into portions of 30 g of dried matter. A total of 30 g of dried fibers was left in 1 L of distilled water to rehydrate, and the disintegration process (Figure 6) was performed following UNE-EN ISO 5263-1:2005 instructions and using a Creusot-loire instrument—adamen-I homargy disintegrator, adding 1 L of distilled water to a previous mixture.



Figure 6. Disintegration process of delignified matter before refining in PFI. Matter from *Phytolacca americana* stem.

The refining process was performed in a PFI laboratory beater mill at 600 and 1200 rpm according to UNE-EN ISO 5264-2:2011. Disintegrated fibers, within a consistency of 10% (UNE-EN ISO 4119:1996), were refined running in tests of 30 s, 1 min, and 2 min, depending on material properties.

Refined fibers were stored for pulp characterization and morphological, mechanical, and optical properties' observation.

The same time test was performed with IAS plant waste, commercially used pulp of Eucalyptus wood, bleached, and submitted to pulping and characterization processes in order to compare properties. In Figures 7–10, we can observe different delignified matter in different steps of the refining process. Leaves and stem matter from different species can be easily distinguished, due their morphological differences.



Figure 7. Delignified fibers from *Arundo donax*.



Figure 8. *Phytolacca americana* stem matter. Different parts of the plant can be observed, stems and leaves.



Figure 9. Refined matter from *Eucalyptus globulus* bark.



Figure 10. Delignified matter from *Tradescantia fluminensis*.

2.3. Pulp Properties

Pulp characterization was developed according to UNE-EN ISO 5267-1:1999 (Schopper–Riegler degree apparatus) to determinate drainability index, expressed as Schopper–Riegler degree (°SR). Results obtained varied from 21 °SR to 82.8 °SR depending on plant matter and refining time.

2.4. Handsheet Preparation and Mechanical Properties of Fibers

Handsheets (Figures 11 and 12) of refined fibers both from commercial pulp and from IAS matter were prepared in the laboratory conventional sheet former according to UNE-EN ISO 5269-1:2005. As indicated in previous studies [39], and due to the paper's condition as a hygroscopic material, handsheets were conditioned under standard climatic conditions at an ambient temperature of 23 ± 1 °C and $50 \pm 2\%$ relative humidity, as prescribed in the standard UNE-EN ISO 187:1990.



Figure 11. Dry handsheet from *Arundo donax* leaves.

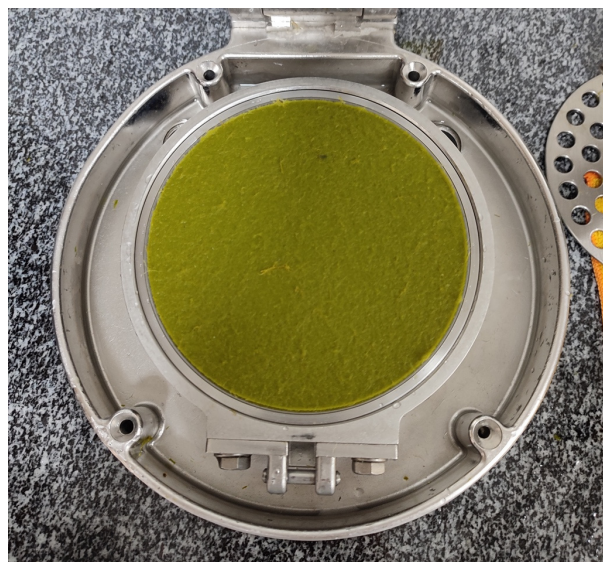


Figure 12. Wet handsheet from *Arundo donax* leaves still in leaf former.

The following properties of handsheets were determined (Table 2): grammage (UNE-EN ISO 536:2012; UNE-EN ISO 3039:2013), thickness, medium thickness, apparent density and apparent volume (UNE-EN ISO 534:2012; UNE-EN ISO 3034:2016), and air permeability according to Gurley (UNE-EN ISO 5636-5:2015).

Table 2. Properties and units developed during experiments.

Properties	Reference Norm	Units
Grammage	UNE-EN ISO 536:2012	g/m^2
Thickness	UNE-EN ISO 534:2012	μm
Specific Density	UNE-EN ISO 534:2012	g/cm^3
Specific Volume	UNE-EN ISO 534:2012	cm^3/g
Drainability	UNE-EN ISO 5267-1:1999	$^{\circ}\text{SR}$
Consistency	UNE-EN ISO 4119:1996	%
Air permeability	UNE-EN ISO 5636-5:2015	$(\mu\text{m}/(\text{Pa}\cdot\text{s}))$
Air resistance	UNE-EN ISO 5636-5:2015	s

2.5. Chemical Composition of Matter

Previous studies and research [17,18] have demonstrated that a high content of anthocyanins and total phenols can be found in compounds extracted from *Arundo donax*, *Tradescantia fluminensis*, and *Eucalyptus globulus*. Additionally, other studies [40,41] have shown that *Phytolacca americana*, beyond the toxicity of its fruits, exhibits anticancer, antioxidant, and antimicrobial properties. The papers produced from this raw material were analyzed using the JEOL 6100 electron microscope located at the CACTI (Scientific and Technical Research Assistance Center) of the University of Vigo, on the As Lagoas campus in Vigo. The surfaces of these papers were mapped (Figure 13), revealing their main compositions: phosphorus (P), carbon (C), and oxygen (O). As these papers were prepared without any additives and processed with standardized water throughout the experiments, it can be concluded that the chemical composition of the raw material remained consistent.

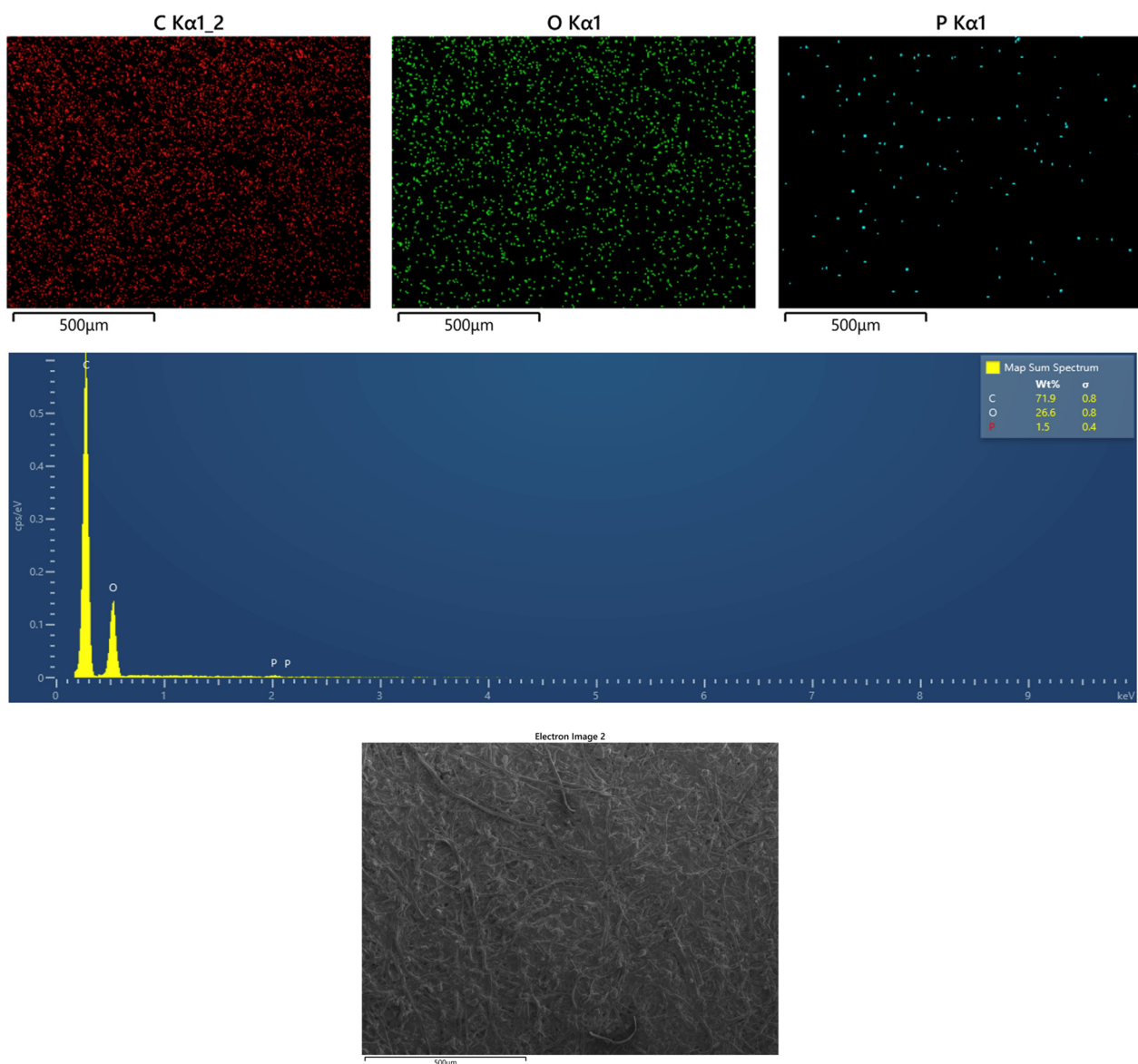


Figure 13. Complete report (map, map spectrum, and referenced imagen, from top to bottom) of elemental composition of the *Arundo donax* (leave) handsheet analysis with SEM visualization. Example of elemental composition of different analyzed matter. Analysis performed at the CACTI installations.

2.6. Optical, Morphological, and Mechanical Properties of Fibers

Optical and morphological properties of fibers (Figure 14) were determined using the JEOL 6100 electron microscope located at the CACTI, in Vigo, Spain. Fiber width was measured by optical images obtained from the optical microscope Nikon SMZ 1500 located at the CACTI laboratory in Vigo, using the software ImageJ 2 (Figures 15 and 16). Results are presented as averaged values (Table 3).

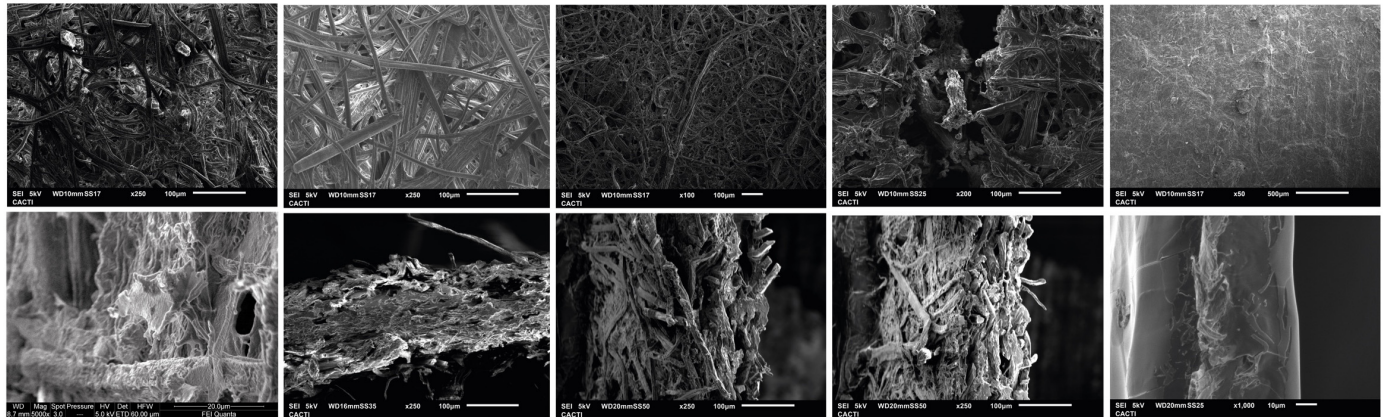


Figure 14. Amplified images of obtained fibers. From left to right, *Arundo donax* Leaves–Stem; *Eucalyptus globulus* Leaves–Bark, *Tradescantia fluminensis*; Top surface image and bottom transversal cut image. JEOL 6100 electron microscope.

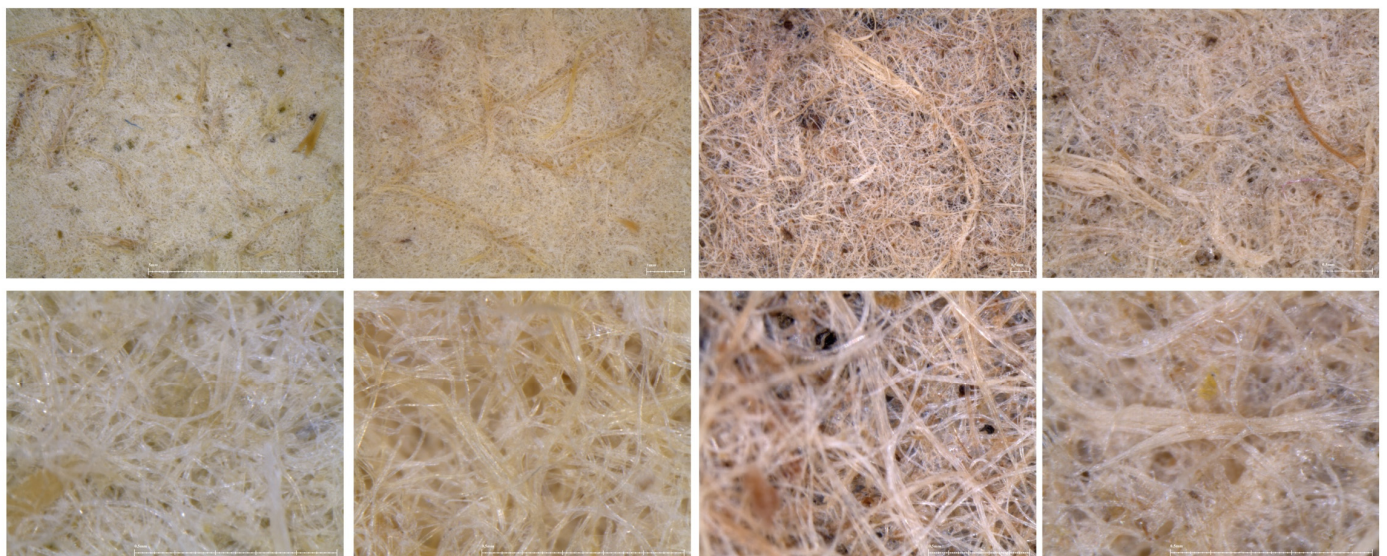


Figure 15. Amplified images of obtained fibers. From left to right, two different amplifications: *Arundo donax* Leaves–Stem; *Eucalyptus globulus* Leaves–Bark. Optical microscope Nikon SMZ1500.

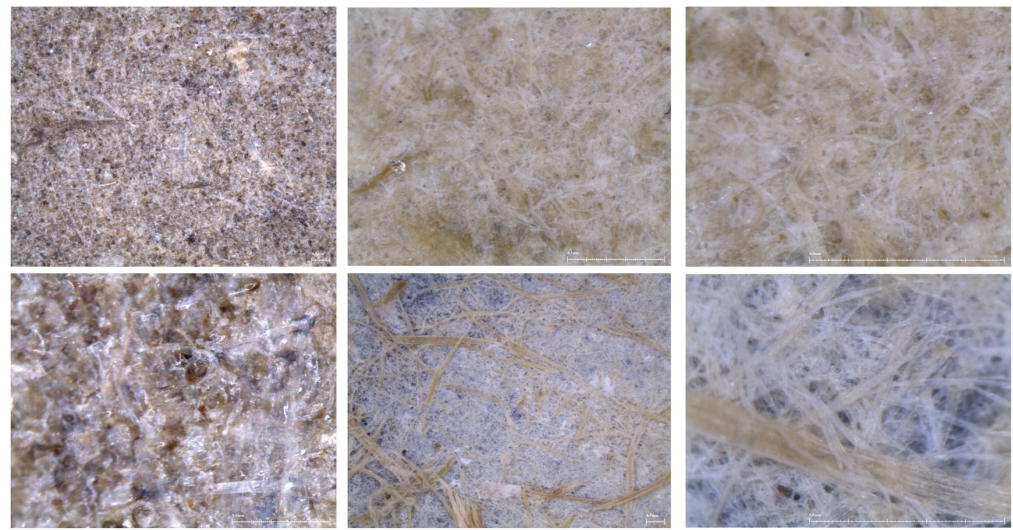


Figure 16. Amplified images of obtained fibers. From left to right, two different amplifications: *Tradescantia fluminensis*; *Phytolacca americana* Leaves–Bark. Optical microscope Nikon SMZ1500.

Table 3. Width of unrefined fibers.

Species	Plant Part	Width (μm)
<i>Phytolacca americana</i>	Leaves	3.9 ± 2.5
<i>Phytolacca americana</i>	Stem	10.9 ± 3.8
<i>Arundo donax</i>	Leaves	10.0 ± 1.6
<i>Arundo donax</i>	Stem	10.5 ± 9.5
<i>Eucalyptus globulus</i>	Leaves	8.4 ± 3.8
<i>Eucalyptus globulus</i>	Bark	9.8 ± 2.8
<i>Tradescantia fluminensis</i>	-	-

2.7. Statistical Analysis

All mechanical and morphological properties were assessed following the ISO standards mentioned previously, incorporating statistical analysis into all measurements. Morphological properties and the fibers' widths were calculated as averaged, whereas mechanical properties were tested in triplicate and performed with three different PFI settings ($30 \text{ s} = 70 \pm 10$, $60 \text{ s} = 120 \pm 10$, and $120 \text{ s} = 240 \pm 10$ loops). Due PFI properties, each loop was equivalent to X10 in new equipment, so, compared with other studies [39], a PFI cycle of 60 s was equivalent to 1200 rpm. In order to avoid possible misunderstandings, PFI information would be shown in seconds (s) instead of rpm. Data shown is the average of the triplicated test. Some tests were unable to be performed due to the technical characteristics of the fibers.

3. Results and Discussion

3.1. Morphological, Optical, and Physic-Mechanical Properties of Produced Handsheets

The morphology of cellulose fibers directly affects different mechanical properties of handsheets [39,42]. The behavior of a handsheet is directly defined by its fiber's composition and properties, such as fiber length and width. In relation to fiber properties and raw matter used for the formation of the sheet, those tests performed with material from woody plants, such as *Eucalyptus globulus*, show different characteristics than those shown by herbaceous plants, *Tradescantia fluminensis* being the most special case. Fibers, as defined by ISO 16065-2, are particles wider than $10 \mu\text{m}$ and longer than 0.2 mm . As shown in Table 3, some of samples collected are effectively wider than $10 \mu\text{m}$, most of them from stems of bark parts of the plant, whereas those analyzed from leaf matter demonstrate to be wide shorter, as in the case of *Phytolacca americana* leaves and *Tradescantia fluminensis*,

where it was not possible to measure fibers due its composition from homogeneous links. As it is explained in study [7], there are usually other smaller particles present in the pulps that are defined as fines and play a filling role but have a minor influence on the paper properties. The result of morphological determinations is shown in Table 3, and those referred to mechanical properties are presented in Table 4.

Table 4. Mechanical properties of refined fibers with different plants, plant parts, and PFI refining times.

	Plant Matter	PFI (s)	Drainability (°SR)	Grammage (g/m ²)	Thickness (μm)	Specific Density (g/cm ³)	Specific Volume (cm ³ /g)	Gurley 300 cm ³ (s)	Air Permeability (μm/(Pa·s))
<i>Phytolacca americana</i>	Leaves	60	63	23.87	71.8	0.33	3.01	43	314.65
		120	21	17.65	139.8	0.13	7.92	<1	-
	Stem	120	34.5	23.53	107.4	0.22	4.56	1.5	9020
<i>Arundo donax</i>	Leaves	60	62	49.79	120	0.41	2.41	25.86	523.20
		120	70	52.03	131.2	0.39	2.52	34.86	388.12
	Stem	60	31	53.47	173.6	0.31	3.25	1.55	8729.03
		120	62	58.74	107.4	0.55	1.83	2.1	6442.85
		120	62	58.74	107.4	0.55	1.83	2.1	6442.85
<i>Eucalyptus globulus</i>	Leaves	60	59	64.56	225.00	0.29	3.48	1.2	11,275
		120	71	59.69	150.80	0.39	2.53	2.9	4665.52
	Bark	60	63	69.64	220.80	0.32	3.17	1.3	10,407.69
		120	64	52.72	179	0.29	3.39	1.5	9020
		120	64	52.72	179	0.29	3.39	1.5	9020
<i>Tradescantia fluminensis</i>	Total	30	65	36.36	108.00	0.34	2.97	262.3	51.58
		60	74.5				*		
		120	82.8				*		
Commercial fiber from <i>Eucalyptus</i>	-	30	18	49.79	140.00	0.36	2.81	<1	-
		60	27.5	53.22	132.00	0.40	2.48	1.65	8200
		120	43	56.70	102.00	0.56	1.79	13.15	1028.00

* No handsheet formation was viable.

There was notice several correspondences in between the mechanical properties of the fibers, common to any biomass origin. With higher PFI times, sheet grammage used to increase, whereas leave thickness tended to decrease. At the same time, specific density increased, whereas specific volume decreased when varying PFI times from less to more.

All the analyzed fibers from the leaf matter demonstrated shorter and thinner fibers, between 3.9 μm (*Phytolacca americana* leave) and 8.4 μm (*Eucalyptus globulus* leave), and the wider was shown in the *Arundo donax* leaves, 10.00 μm. On the other side, the fibers from the barks of stems were demonstrated to be thicker, from 10.90 μm (*Phytolacca americana* stem) and 10.50 μm (*Arundo donax* stem) to 9.8 μm (*Eucalyptus globulus* bark).

It has been presented and confirmed that raw matter used for sheet formation is related to its properties. Woody species, such as *Eucalyptus globulus*, demonstrated the production of thicker handsheets (Figures 17 and 18) independent from the raw matter used, varying from 225 μm (leaves) and 220 μm (bark) in comparison with those elaborated from herbaceous matter such as *Tradescantia fluminensis* that showed 108.00 μm or *Phytolacca americana* leaves (71.8 μm), whose results showed that the thinner handsheet elaborated on was directly linked with its long fibers. The obtained values were comparable to other similar studies with IAS applied to the paper industry [7,38,39].

As it was demonstrated by the tests, wider and longer fibers used to be associated with porous, less uniform, and coarse paper structures but, at the same time, showed higher strength properties [43].

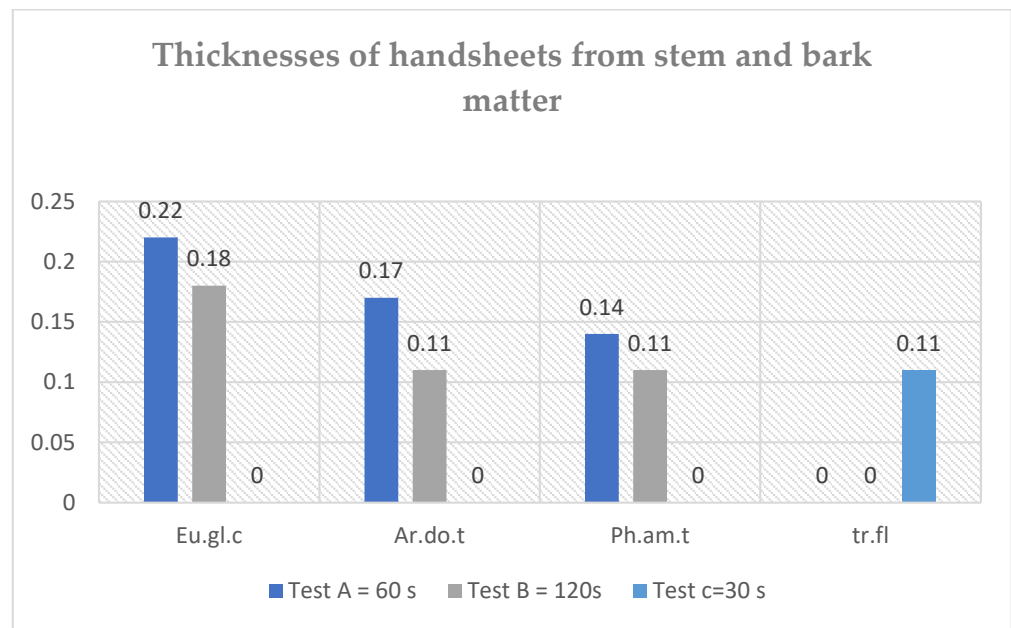


Figure 17. Thicknesses of handsheets elaborated from stem or bark raw matter in functions of different PFI (Test A; Test B; and Test C) times for four analyzed species.

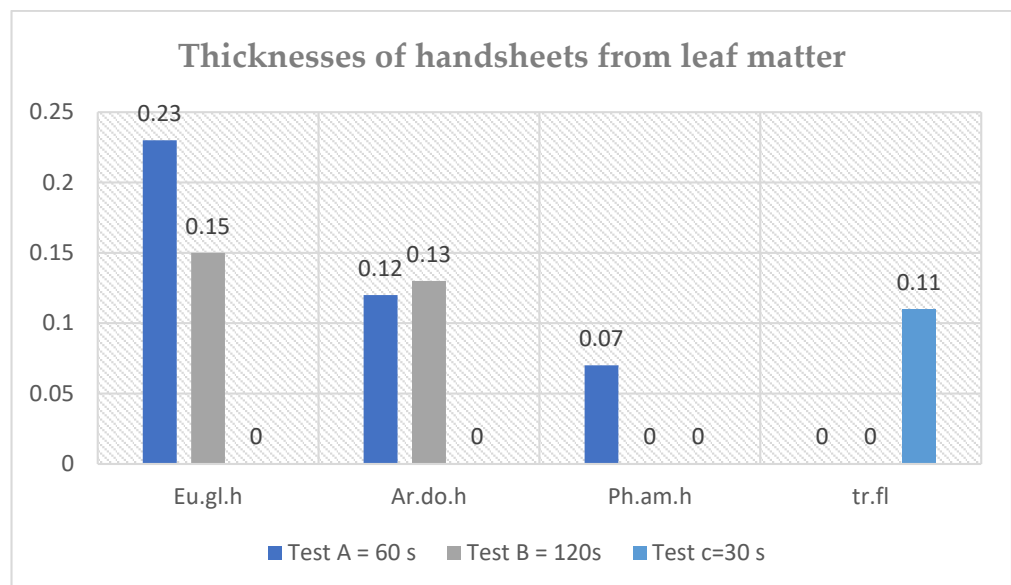


Figure 18. Thicknesses of handsheets elaborated from leaf raw matter in functions of different PFI (Test A; Test B; and Test C) times for four analyzed species.

Paper thicknesses (presented in Figures 17 and 18) were directly connected with air permeability properties, demonstrating that handsheets made from leaves or herbaceous matter presented higher Gurley times. Four tests performed with leaf matter had relatively high Gurley times in comparison with those analyzed from stem or bark matter. *Phytolacca americana* leaf matter presented a Gurley time of 300 cm³ in 43 s, whereas the tests run with stem matter were <1 s or 1.5 s, depending on pulp refining times. *Arundo donax* leaves also presented higher Gurley times than stem matter, varying from 25.86 s (60 s PFI) or 34.86 s (120 s PFI) to 1.55 s (60 s PFI) or 2.1 s (120 s PFI). In the case of *Eucalyptus globulus*, as unique woody matter, it presented lower Gurley times for both leaves and bark matter (1.2 s; 2.9 s leaves and 1.3 s; 1.5 s bark).

It is necessary to present the *Tradescantia fluminensis* results as the most relevant in relation to air permeability. Due its morphological properties, this species demonstrated higher Gurley times with great differences, and also developed shorter PFI times (30 s), reducing yields in relation to the refinement of time properties. As shown in Table 4, *Tradescantia fluminensis*, for 30 s PFI refining time, presented a Gurley time of 262.3 s.

The performed test demonstrated a relation between Schopper–Riegler refining degrees of pulp matter and Gurley time. It can be concluded that generally higher Schopper–Riegler degrees demonstrated higher Gurley times and smaller air permeability values in handsheets. The Schopper–Riegler test was designed to provide a measure of the rate at which a dilute suspension of pulp might be dewatered [39]. The relation of the drainability test is presented in Figure 19, and the relation of air permeability is presented in Figure 20. After running the test, it was concluded that Schopper–Riegler degrees higher than 72 were not possible to perform the handsheet test. It is the case for the test developed with *Phytolacca americana* at more than 60 s at PFI, and for those performed with *Tradescantia fluminensis* at 60 s and 120 s, that they were needed to reduce PFI times to 30 s.

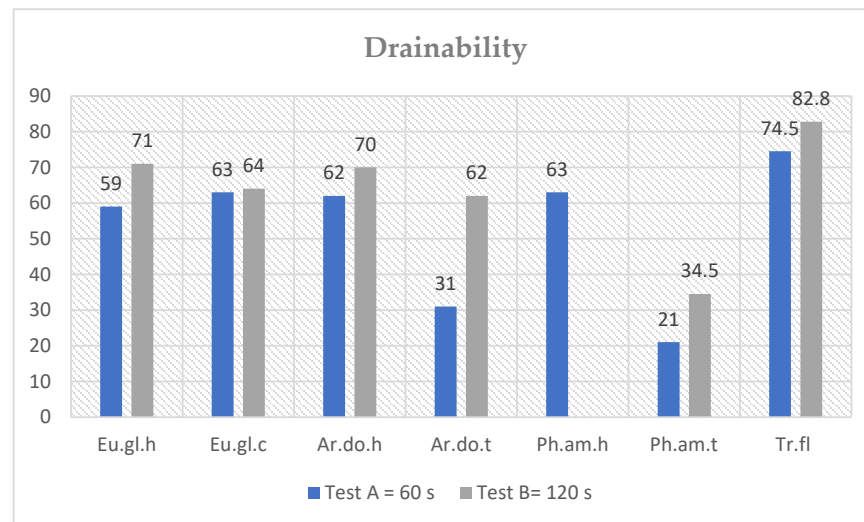


Figure 19. Drainability (°SR) in function of two different PFI (Test A; Test B) times for four analyzed species and matter (.h from leaves, .c for from bark, and .t from stems).

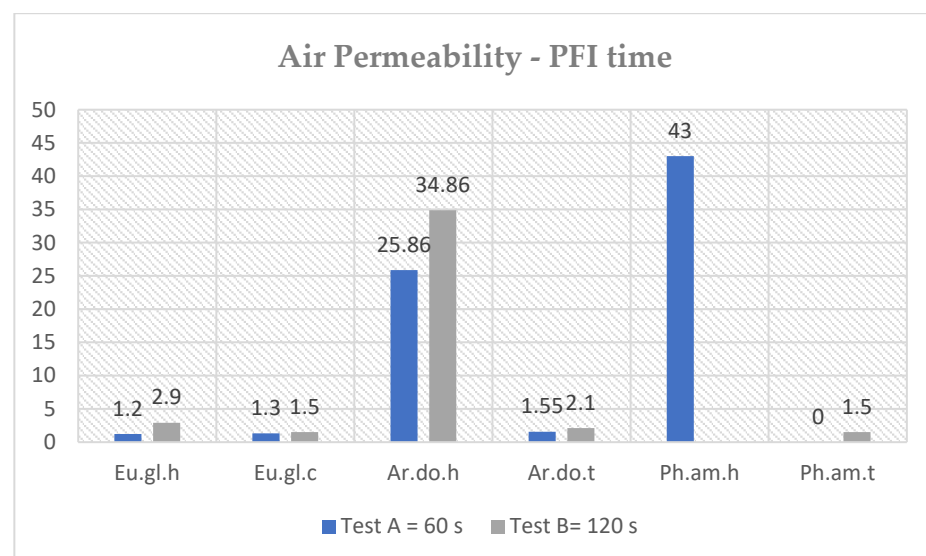


Figure 20. Air permeability in (s) in function of two different PFI times (Test A; Test B) for four analyzed species and matter (.h from leaves, .c for from bark, .t from stems).

Upon conducting a comprehensive examination of morphological characteristics, it becomes imperative to delve into the pulp properties, specifically focusing on drainability, to gain deeper insights into material behavior. Pulp derived from leaves exhibits elevated Schopper–Riegler ($^{\circ}\text{SR}$) degrees when subjected to shorter PFI refining times. Typically characterized by shorter and finer fibers compared to those sourced from bark or other plant parts, this raw material historically contains a greater proportion of fines. Fines, being associated with larger surface areas, exhibit an enhanced capacity for swelling, resulting in quicker retention within the sieve of the Schopper–Riegler apparatus. This phenomenon leads to elevated $^{\circ}\text{SR}$ values, consequently yielding more favorable mechanical properties [39].

3.2. Comparison of Properties with Commercial-Bleached Eucalyptus Pulp Handsheets

The analyzed physical–mechanical and structural properties of paper (grammage, thickness, density, apparent volume, and air permeability) have a significant impact on its quality and suitability for different applications. For this reason, it is relevant to understand paper properties in order to find suitable opportunities for innovation. Below, we present the relationship between the properties of papers made from different parts of the selected Invasive Exotic Species, in comparison to those of paper made from commercial Eucalyptus pulp.

As is well-known, a higher grammage is generally associated with thicker and more durable paper, which is important for applications requiring strength and durability. However, excessively high grammage can make the paper stiff and challenging to fold, which may be undesirable for certain applications like printing. Moreover, thicker paper generally has a higher printing capacity and a better appearance, which is crucial for high-quality printing applications and influences paper stiffness and strength. Furthermore, a higher specific density is usually linked to more compact and durable paper, and, on the other side, a higher specific volume typically indicates that the paper is more porous and less dense. Both properties can influence ink absorption in printing applications and the tactile feel of the paper.

The Eucalyptus properties (performed test results can be observed in Table 5) of high pulp yield, high wood density, excellent fiber quality, and good handsheet properties make this species one of the most important hardwood species used in the paper industry [44]. In Galicia, Spain, it is possible to find broad areas destined to grow Eucalyptus monocultures, whose final objective is use within the paper and wood industries.

Table 5. Commercial fiber made handsheets mechanical properties. Test A is PFI the time of 60 s; Test B is the PFI time of 120 s; and Test C is the PFI time of 30 s.

Properties	Test A	Test B	Test C
Drainability ($^{\circ}\text{SR}$)	27.50	43.00	18.00
Grammage (g/m^2)	53.22	56.70	49.79
Thickness (μm)	132.00	102.00	140.00
Specific density (g/cm^3)	0.40	0.56	0.36
Specific volume (cm^3/g)	3.48	1.79	2.81
Air resistance-Gurley 300 cm^3 (s)	1.65	13.15	<1
Air Permeability ($\mu\text{m}/(\text{Pa}\cdot\text{s})$)	8200.00	1028.00	-

In recent years, commercial pulps have been undergoing changes in their composition, despite Eucalyptus remaining one of the most commonly used species as a source of cellulose in Europe. Despite the proven properties of wood-derived cellulose, other studies [45] propose cellulose extracted from non-wood species as a raw material for the production of commercial paper, yielding different properties through its use.

This is the case with *Tradescantia fluminensis*, which, unlike the commercial pulp used in this study, provides higher Schopper degrees and consequently lower air permeability (and higher Gurley times) with shorter refining times (with a refining time of 30 s, we obtained 65 °SR and 262.3 s of air passage resistance, whereas, with the commercial pulp used, we obtained 18 °SR and Gurley times of less than 1 s). This, as shown in Figure 21, can also be observed in the test sheets made from *Phytolacca americana* leaves and *Arundo donax* leaves, concluding that leaf matter demonstrates great potential in relation to air permeability and air resistance properties for shorter refining periods.

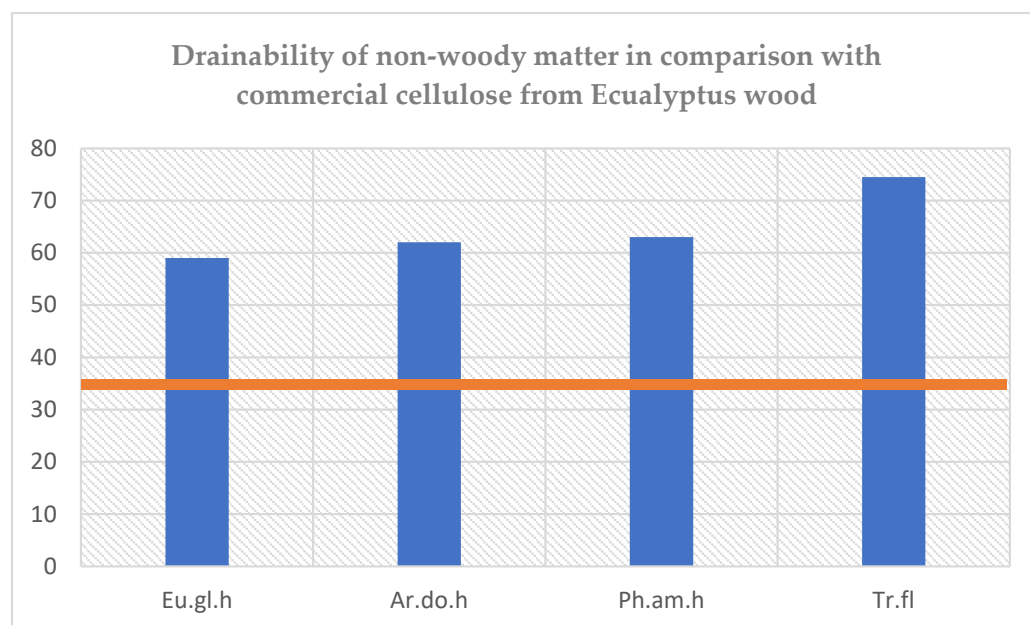


Figure 21. Schopper (°SR) degrees for non-wood raw matter cellulose extracted handsheets in comparison with commercial pulp from Eucalyptus (orange line) with a PFI time of 60 s.

It is relevant to highlight that unbleached pulps delignified from crafted and less-contaminated processes allow us to obtain competitive properties, obtaining values, on occasion, similar to those obtained from bleached commercial fibers. This discovery is considered significant because one of the major sources of pollution in the paper industry, during the pulping process, is the bleaching of fibers, which is sometimes unnecessary for the intended purpose of the paper under development.

Specific densities demonstrated as inferior for the same refining times (Figure 22), whereas specific volume values were higher (Figure 23). Variations in grammage and thickness can be observed in Figures 24 and 25. Depending on the sheet's raw matter origin (leave or stem/bark), values are higher or lower. For grammage, sheets developed with leaves were demonstrated to have smaller values than those performed with commercial fibers, and this was on the contrary for handsheets created with bark or stem matter, except for *Phytolacca* stems and *Eucalyptus* leaves. Regarding the thickness properties, leaf values were maintained as lower than commercial fibers, except for *Eucalyptus* leaves, and the thicknesses remained higher for the barks of stem biomasses. These comparisons were performed with PFI times of 60 s, and, for this reason, *Tradescantia fluminensis* was not included due its impossibility of sheet formation with PFI times higher than 60 s.

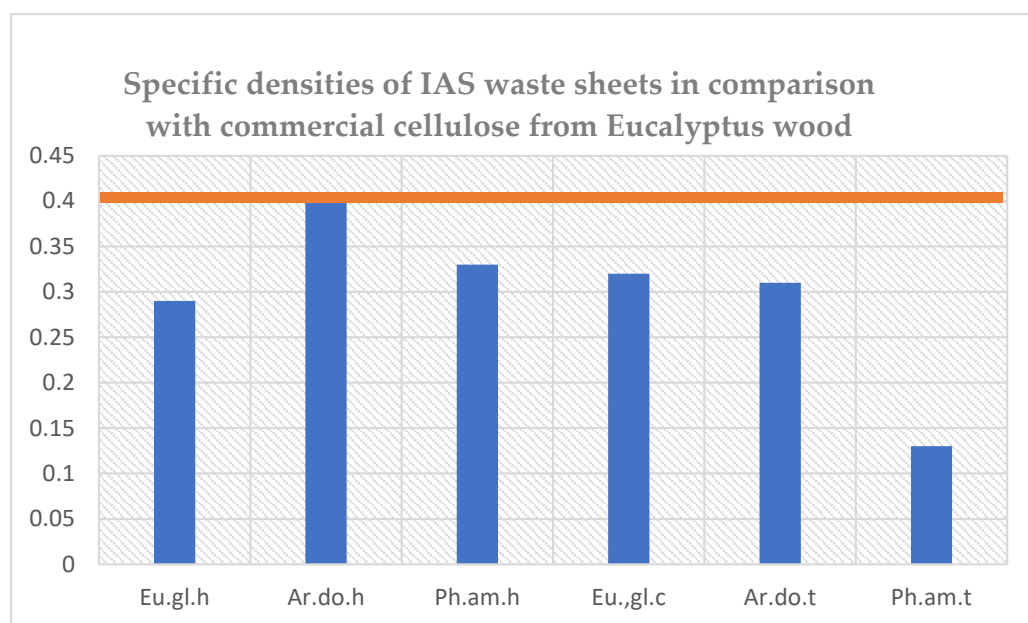


Figure 22. Specific densities (g/cm^3) of IAS raw matter (.h from leaves, .c for from bark, and .t from stems) cellulose extracted handsheets in comparison with commercial pulp from Eucalyptus (orange line), with a PFI time of 60 s.

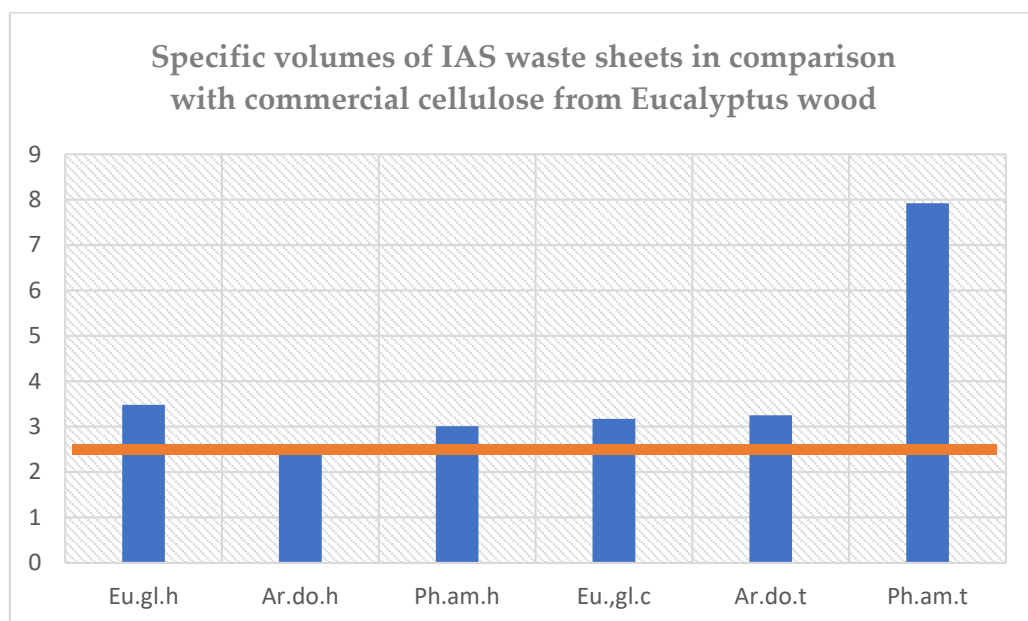


Figure 23. Specific volumes (cm^3/g) of IAS raw matter (.h from leaves, .c for from bark, and .t from stems) cellulose extracted handsheets in comparison with commercial pulp from Eucalyptus (orange line), with a PFI time of 60 s.

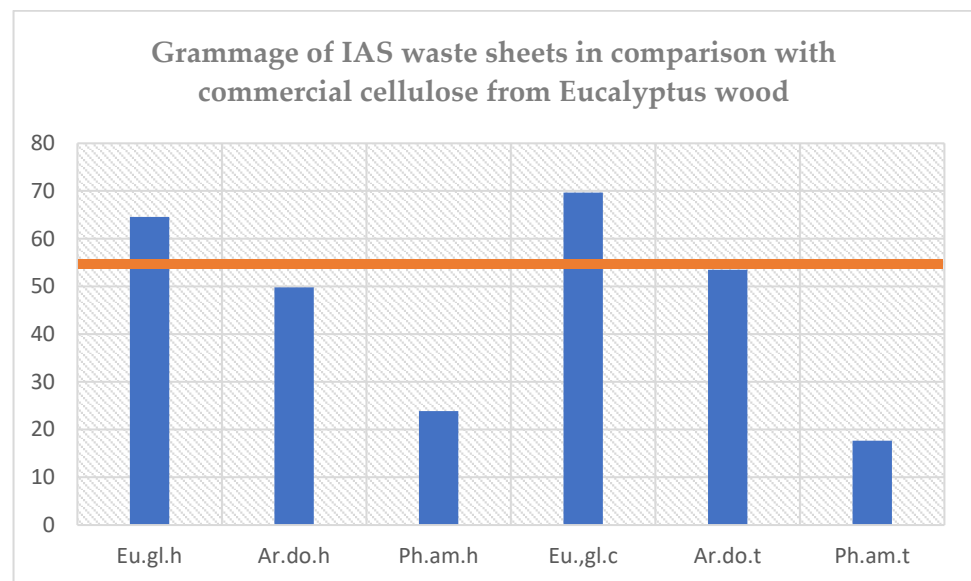


Figure 24. Grammage (g/m^2) of IAS raw matter (.h from leaves, .c for from bark, and .t from stems) cellulose extracted handsheets in comparison with commercial pulp from Eucalyptus (orange line), with a PFI time of 60 s.

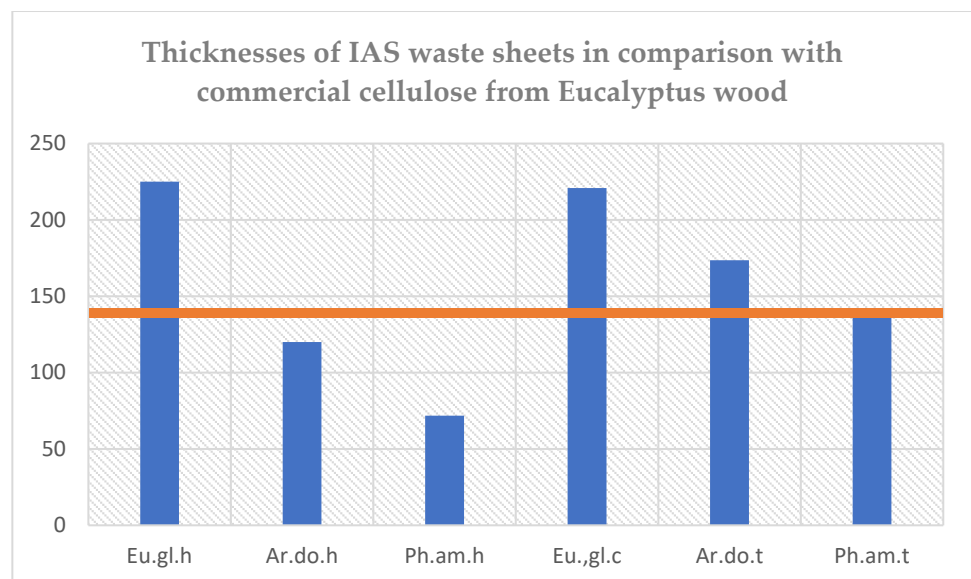


Figure 25. Thicknesses (μm) of IAS raw matter (.h from leaves, .c for from bark, and .t from stems) cellulose extracted handsheets in comparison with commercial pulp from Eucalyptus (orange line), with a PFI time of 60 s.

4. Conclusions

Selected IAS fibers were demonstrated to have potential physical and optical capacities for developing papermaking, specially focused for artistic and graphical purposes. The developed paper mechanical and visual properties, such as textural composition, visual color degradation, among others, showed great potential for artistic ends, becoming, for example, a possible tool for environmental awareness communication. Although the analysis of the specific uses of these species in the commercial paper industry was not one of the objectives of this study, but rather to verify their suitability for paper production, it has been confirmed that there are intriguing possibilities for the industry, and these should be thoroughly investigated in future studies.

Due to its characteristics in relation to permeability and weight–grammage relation, sheets made from *Tradescantia fluminensis* showcased the most interesting and unseen results. There’s either poor or no literature regarding this plant species for papermaking, and, due to its huge capacities for adaptability and expansion, the reutilization of it seems to have great potential in the paper industry for diverse uses. Sheets created with *Tradescantia fluminensis* were demonstrated to have relevant air resistance. *Arundo donax* and *Phytolacca americana* sheets created with stem material presented similar capacities than commercial bleached eucalyptus fibers, but its visual potential stood out due to its textural outcomes. This makes them suitable for the creative field, filling the artistic gaps in the industry. On the other hand, handsheets elaborated on from leaf material demonstrated great capacities in relation to air permeability, demonstrating higher results than commercial fibers. Finally, bark and leaves from *Eucalyptus globulus* sheets demonstrated similar properties with commercial pulps, presenting potential for its reintroduction into the paper industry.

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