

## Chapter

# Integration of Waste from Invasive Alien Species *Phytolacca americana* with Eucalyptus Commercial Pulp for Papermaking: A Review on the Evolution of Physic-Mechanical and Chemical Paper Properties

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## Abstract

The paper industry contributes significantly to the transformation of native forests into monocultures of fast growing exotic species such as *Eucalyptus globulus*, reducing biodiversity. This expansion, driven by the demand for cellulose-based products, threatens ecosystems by displacing native flora and causing soil impoverishment. To address these issues, the study explores the possibility of reusing waste fibers obtained after cleaning areas affected by a major presence of invasive species such as *Phytolacca americana*, as a sustainable alternative to traditional wood fibers in paper production. The research focused on Ribadumia riparian forest in Galicia, Spain, and involved the entire process of obtaining and processing *Phytolacca americana* pulp. It assesses the physical and mechanical properties of the resulting paper, using advanced techniques such as SEM visualization and physical paper tests. Results confirmed the suitability of *Phytolacca americana* fibers for paper production, with certain properties showing improvement and/or maintenance over those from commercial paper. Blends of *Phytolacca americana* and commercial bleached *Eucalyptus globulus* pulp demonstrated comparable quality, suggesting the feasibility of replacing commercial fibers like *Eucalyptus globulus* with those derived from the waste of invasive species. This approach could not only contribute to reduce monocultures and promote biodiversity, but to apply zero-waste dynamics in usually pollutant industries.

**Keywords:** invasive alien species, paper production, biomass reutilization, non-woody pulp integration, biodiversity loss

## 1. Introduction

Paper and pulping industries are extremely related to environmental and ecosystem health. The paper industry is not only the fifth sector most pollutant, generating

an amount of 220–380 m<sup>3</sup> of highly colored and potentially toxic wastewater [1], but it is responsible for most monoculture production, consuming 40% of the world's commercially cut timber. Apart from this, paper production remains one of the most relevant industries globally, acquiring a total amount of 2.519.089 € in past 2018 only in Spain, and as stated by the As Papel Association, in 2022, Spain's overall production reached 6.3 million tons, with 1.5 million tons designated for pulp and 6.3 million tons for paper and cardboard. This situation positioned Spain as sixth in paper and fifth in pulp production within the EU (European Union). According to preliminary CEPI (Confederation of European Paper Industries) reports in 2021, the paper industry has increased by 5.8% in relation to the previous year [2].

Those wicked situations contribute to the deterioration of natural ecosystems surrounding factory mills and pulpwood plantations. Paper mills are usually located near wood plantations in order to decrease costs; natural forests were usually used for papermaking industry in the past, but, nowadays, the use of natural forests is being substituted by plantations of fast-growing species, usually exotic, such as *Eucalyptus* and Pines. Planted forests were, in 2014, 7% of the total global forest area, or 264 million ha [3].

According to the Annual Forest Statistics of 2018, 33% of timber is destined to pulp production, the highest proportion of wood commercial uses. In comparison, in the case of the sector, 38% of wood is destined for bioenergy, and 21% for pulp production [4]. At the north of Spain, specifically in Galicia, there is a volume of timber areas of 125m<sup>3</sup>/ha in front of 189 m<sup>3</sup>/ha of forest surface. Compared with this, the south of Spain presents an average of 20 m<sup>3</sup>/ha – 50 m<sup>3</sup>/ha [4]. A third part of total forest plantation in Galicia is destined to commercial deforestation, regarding the felling of tree areas for commercial and industrial purposes and interests. The three principal arboreal species in Spain are from the *Pinus* family, but the fourth one, even exotic, is *Eucalyptus globulus*, occupying 79.645.351 m<sup>3</sup> c.c. in all Spanish surface [5], part of the forest area destined to production, an 8% of the total forest area, specifically 619.918 ha.

*Eucalyptus globulus* plantations proliferation in Galicia, Spain, are mainly derived from ENCE (Energy and Cellulose Paper Mill), Pontevedra's paper mill, which in 2022 spent 236.000.000€ in timber from individuals [6], covering 95% of demand needs. Other studies demonstrated that a third part of the Galician Forest has been planted by humans, with commercial ends.

*Eucalyptus globulus* species are not classified as invasive exotic species in Spain, according to the Invasive Exotic Species Catalog. However, this exotic species has demonstrated expansive behavior altering local ecosystems. Among other conditions, its leaf's chemical composition, which intervenes in soil, increasing its pH and avoiding biodiverse flora proliferation [7], derives in preventing local species from growing. Even nowadays, several studies [8, 9] demonstrated high possibilities for *Eucalyptus globulus* industry by-products (such as leaves and bark) reutilization for antioxidant extraction and papermaking; main problem remains in high commercial demands that are translated into monocultures incrementation [6].

Furthermore, the problematic relationship between monocultures and biodiversity flora maintenance, in addition to forest urbanization and anthropogenic factors as agriculture and land management, usually results in ecosystem diversity loss, favoring exotic and invasive species proliferation [10]. Local vegetation in riparian areas is being naturally substituted by invasive species such as those selected for this study, *Phytolacca americana* [11]. The impact of invasive alien species in natural ecosystems

disturbing its balance through nutrient and resource competition, disease transmission, and intertwining in soil and light conditions, provoking the reconfiguration of the ecosystem functioning [2, 12]. The major presence of invasive plant species in managed environments tends to have consequences in the ecological integrity of the ecosystem, altering the natural composition of species and habitat [13, 14].

According to the European Commission, 41 plant species pose a threat to European natural ecosystem's biodiversity. In Regulation (EU) 1143/2014, Invasive alien species are presented as one of the main threats to biodiversity and related to ecosystem services, which are being intensified by global trade, transport, tourism, and climate change, consisting of a cross-border challenge affecting the whole Union [11]. When referring to invasive alien species, it is relevant to confirm anthropogenic introduction and naturalization in different ecosystems than its original habitat; by human means, these species were usually introduced for cultivation or for ornamental means and derived into ecosystem degradation [11]. In affected ecosystems, rapid restoration and mitigation of Invasive species is crucial for flora maintenance; for this reason, several management activities are being performed all around affected areas, such as the one selected for this study, in Ribadumia, Galicia. Derived from this management initiative, promoted by the city council and governmental decisions, amounts of fiber waste are being generated, which were suitable to be reintroduced into production systems.

In order to help mitigate and reduce paper industry contradictions with ecosystem biodiversity and maintain of ecological integrity of natural forests, this study proposes the reutilization of invasive alien species *Phytolacca americana* plant waste, after cleaning initiatives, as a substitution for *Eucalyptus globulus* commercial pulp in pulping and papermaking industry. The substitution of first raw materials for secondary raw matter helps mitigate waste production and waste burns and reduce price volatility, availability, and import dependency [15].

The need for creating and designing sustainable products is an increasing concern, becoming one of the most relevant issues nowadays. According to the European Commission Circular Economy Action Plan [16], the anticipated global consumption of materials like biomass, fossil fuels, metals, and minerals is set to double within the next 40 years. Concurrently, annual waste generation is forecasted to surge by 70% by 2050 [17].

The selected area of study and recollection for this research was the riverbank area of Umia River in Ribadumia, Galicia, Spain, which is affected by the presence of several invasive alien species due to anthropogenic factors [10]. *Phytolacca americana* (**Figure 1**) has been identified as one of the most present alien species in riparian forests, and its controversial effects have been studied by experts [18].

Selected species *Phytolacca americana* is a perennial plant of the Polygonaceae family, native to Eastern North America, commonly known as American pokeweed, poke sallet, or inkberry. It is a large herbaceous species, reaching up to 3 meters. Its stem (**Figure 2**) is cylindrical, fleshy, and hollow inside, with purple or red spots, having a woody constitution at the base; it is easily identifiable due to its reddish/fuchsia coloration [10]. The root of *Phytolacca* is thick, with multiple heads and a strong system of taproots [19]. The leaves are alternate, simple, lanceolate, and bi-tonal, darker on the upper side. The maximum length of the leaves is 25 cm. Its fruit is fleshy and intensely purple, highly toxic, like all parts of the plant [20].

*Phytolacca americana* is one of the most common herbaceous invasive alien species in Mediterranean areas [21]. Nationally, it is included under Royal Decree 630/2013 in the Spanish Catalog of Invasive Alien Species; at the European level, it is regulated by Regulation (EU) 1143/2014 of the European Parliament and of the Council of 22



**Figure 1.**  
(left) *Phytolacca americana* in the riverbank area of Umia river, Spain.



**Figure 2.**  
(right) Detail of stem and fruit of *Phytolacca americana*.

October 2014 on the prevention and management of the introduction and spread of invasive alien species; and internationally, under the Convention on Biological Diversity (CBD) 1992, as well as the Convention on the Conservation of European Wildlife and Natural Habitats (Bern Convention, 1979) (Life, Med, WET Rivers, IAS, PHYAME/EPI/FL032).

Currently, the distribution of this species has significantly expanded, being naturalized in various regions of the world beyond its natural habitat in North America [19]. From Canada to Argentina in the Americas or since the 17th century in Europe, where it began to be cultivated around the Mediterranean. References to the cultivation of this species in areas of North Africa and Southern Europe date back to the mid-17th century [19]. Similarly, this plant has naturalized in other parts of the world, from Turkey, Asia, India, China, Taiwan, Japan, Indonesia, and Australia, to New Zealand. It is one of the most widely distributed species globally and is, in numerous locations, considered an IAS [19, 21].

The plant's life cycle in the Mediterranean climate occurs between April and October, disappearing during the colder months. The plant begins to sprout as the

average temperatures rise after winter, and the first plants can be found in April or May [19].

According to [19], it can be found in uncultivated vineyards, orchards, cultivated fields, ruderal areas, or even urban areas. The fruits ripen during the warmest seasons, and it is from the last months of autumn and the first of winter that the plant disappears completely, although the root remains from 1 year to the next. It can be found growing annually in the same areas [21]. In the riverbank area of Ribadumia, it can be found isolated from other individuals of its species, growing in the middle of other species such as *Tradescantia fluminensis*. But it can also be found in residual spaces between agricultural land, forming big groups of individuals dominating the spaces.

In this study, fibers from *Phytolacca americana* were mixed with commercial bleached pulp from Eucalyptus, commonly used in the paper industry. Raw matter was donated to the Forestry Engineering School of the University of Vigo by the ENCE Research Laboratory in Marin, Pontevedra.

Therefore, the present research aims to analyze the potential of fibers from the invasive alien species *Phytolacca americana* as a replacement for commercially used fibers derived from monocultures of trees such as *Eucalyptus globulus*. By focusing on the properties of laboratory-tested handsheets, we examine various proportions of raw material, starting with 100% *Phytolacca americana* and progressing through a series of mixtures—100%, 90%, 70%, 50%, 30%, 10%, and 0% in relation to *Eucalyptus globulus* commercial pulp. This stepwise approach allows for a comprehensive comparison of the resultant paper sheet properties in terms of air resistance, drainability, grammage, and thickness. The findings from this research aim to provide valuable insights into the feasibility of using *Phytolacca americana* fibers as a sustainable alternative to traditional monoculture-sourced fibers for the papermaking industry and extended to printing applications. Thereby contributing to the reduction of the environmental impact associated with controversial commercial pulp origins, such as monocultures, this research initiative search to advance into biodiversity maintenance and vegetal waste management in areas affected by the presence of invasive alien species.

## 2. Materials and methods

For this study, it has been used and applied specific instruments and processes. As follows, it is possible to find the materials and methods involved in its development.

### 2.1 Harvesting and delignification process

Harvesting of raw matter from *Phytolacca americana* was carried out in June of 2022 in a specific area of 2 km in the river Umia riparian forest space between Cabanelas and Ribadumia, Spain. In order to proceed with the de-lignification process, raw matter was divided into leaves and stems, cut into pieces of a maximum of 20 cm, and dried in the oven at the Wood Chemistry Laboratory at the Forestry Engineering School at a temperature of 105°C for 24 h. After dehydration, stems and leaves were weighed and stored in a cooking pot of 12 L of capacity. A 20% of its weight was added in Caustic SOSA (NaOH) content, and distilled water was added until full covering of the material, an average quantity of 5 L. As observed in previous



**Figure 3.**

From left to right, delignified raw matter from *Phytolacca americana* (1) leaves, (2) small branches and leaves mixture, and (3) stem material.

research [22, 23], optimized times and quantities are a ratio of 5:1 liquid-to-solid mix and 90 minutes of cooking time. Temperature was selected by maintaining the content at the boiling point. Internal conformation of matter, which presents strong and long fibers in the stem area, suggested the addition of 30 minutes extra for delignification process, performing a total time of 120 min at boiling temperature.

After delignification, the black bleaches resulting from the process, were filtered and stored in special containers. It is possible to neutralize its pH with sawdust in order to use it as compost material for further occasions, as we can observe in **Figure 3**, raw delignified matter presents completely different structures when referring to leaves and stem material. While stems keep their original strength, leaves are almost decomposed in a homogeneous mix.

Once raw matter was cleaned with distilled water, it was dried at the hoven during 24 h at 105°C, for further pulping and refining processes.

Previous studies performed with *Phytolacca americana* demonstrated that more suitable results of papermaking were obtained with stem material, so in this study, just stem matter was used for blending with commercial pulp due to similar conditions. In the publication [22], *Phytolacca americana* leaves pulp material obtained a drainability degree of 63°Schopper-Riegler (SR) and stem matter oscillated in between 21°SR and 34.5°SR depending on refining times (60 seconds or 120 seconds). On the other hand, commercial pulp with 60 seconds of refining time obtained 27.5°SR or 43°SR for 120 seconds, which is closely to the drainability obtained with *Phytolacca* stem material. It is for these reasons that it has been decided for this study to specifically use raw matter from the *Phytolacca* stem, as it appears to demonstrate similar properties and behavior with *Eucalyptus globulus* commercial pulp.

## 2.2 Pulping and refining process

Stem material from *Phytolacca americana*, once dried, was divided into small portions of 30 g and rehydrated with 1 L of distilled water, according to UNE-EN-ISO 5263-1:2005 [24] indications for commercial pulp. Then, commercial pulp dried material was submitted in the same process, rehydrated with 1 L of distilled water. The refining process was performed separately.

First, unbleached matter from *Phytolacca americana* stem was disintegrated in a laboratory disintegration beater, obtaining a consistency of 10%, ready to be refined, following indications set up in UNE-EN ISO 4119:1996 [25]. Mixture was refined for 2 minutes in a PFI laboratory beater mill at 600 and 1200 rpm according to UNE-EN-ISO 5264-2:2011 [26]. Refined material was then weighed, and distilled water was added until 300 g of material was reached.

The same process was performed with commercial bleached pulp from Eucalyptus. First, disintegrated and then refined for 2 minutes. Resultant pulp was measured, and the matter was adjusted with distilled water until obtention of needed quantities.

In order to perform efficient laboratory sheets and physic-mechanical tests, raw matter was divided into proportions of a mixture of 10%, 30%, 50%, 70%, and 90% of raw matter from *Phytolacca* in relation to commercial pulp material. After calculation, the raw matter was mixed and stored for pulping test development before laboratory sheets formation. Related information with 100% *Phytolacca americana* handsheets and 100% commercial pulp handsheet properties obtained in previous studies [22] was used to have a global vision of physical characteristic evolution.

### 2.3 Physic-mechanical properties analysis of pulp and Handsheets

After performing the refining process separately, each pulp matter was weighed to obtain its corresponding part in order to perform each test. To achieve each proportion quantity for optimizing 30 g of raw matter from *Phytolacca* and 30 g of raw matter of commercial pulp the following equation was calculated:

$$Q = \frac{\text{Percentage } n \times \text{raw matter mass}}{(\Sigma \text{ percentage})} \quad (1)$$

where

$Q$  is the raw matter quantity to be used.

Percentage  $n$  is the relative percentage to be calculated.

Raw matter mass is 30 g of *Phytolacca* or commercial pulp.

$\Sigma$  percentage is the summation of all percentages to be calculated.

The result is presented as grams of mass to be used in each mixture test in order to maintain the final mass constant according to the study [25]. The proportion of distilled water added to the experiment has been adapted to maintain proportions of consistent pulp dissolution. The refining time was 120 seconds with an initial raw matter of 30 g. The following pulp quantity for the mixture of *Phytolacca americana* and commercial pulp are presented in **Table 1**.

The calculated pulp mixture was adapted to consistency needs following UNE-EN-ISO 5267-1:1999 [27] in order to perform the drainability Schopper-Riegler (°SR) test. Prepared samples of selected proportions were disintegrated and reformulated with distilled water to obtain a consistency of 0.5% to perform the test with the Schopper-Riegler apparatus.

#### 2.3.1 Schopper-Riegler drainability property and calculation process

As specified in Ref. [27], the drainability index refers, in the context of papermaking, to the feasibility and facility of how water can be removed from a fiber suspension. To this end, the calculation of the drainage index involves measuring the

| Mixture proportion | Pulp quantity for mixture (g) |                 |
|--------------------|-------------------------------|-----------------|
|                    | <i>Phytolacca americana</i>   | Commercial pulp |
| 10–90%             | 1.2                           | 10.8            |
| 30–70%             | 3.6                           | 8.4             |
| 50–50%             | 6                             | 6               |
| 70–30%             | 8.4                           | 3.6             |
| 90–10%             | 10.8                          | 1.2             |

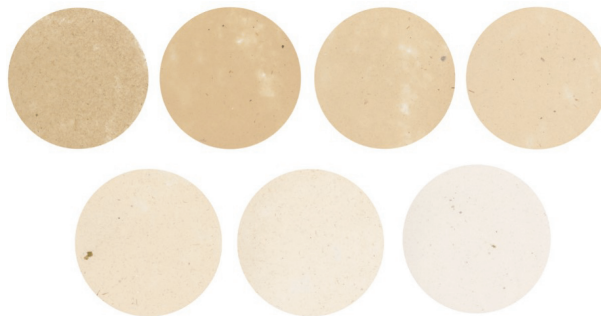
**Table 1.**

*Pulp quantity to be used for mixture proportions for pulping tests.*

drainage capacity of a fiber cake formed on a specific apparatus called Schopper-Riegler, which measures in this same scale. Results are expressed in Schopper-Riegler degrees (°SR). A higher index means a more tighter surface, so a denser and compact fiber. On the contrary, lower °SR used to imply longer and thinner fibers with bigger holes in between.

After drainability tests, laboratory sheets were made with each pulp mixture. A conventional sheet former, ubicated at the Wood Chemistry Laboratory of the Forestry Engineering School, with a diameter of 16 cm, was used with blended pulps, following UNE-EN-ISO 5269-1:2005 [28] indications, and ambient conditions were measured and set according to UNE-EN-ISO 187:1990 [29] standards. In order to achieve an average and significant result, each test was performed in triplicates. Obtained handsheets can be observed in **Figure 4**, where it is possible to notify color and texture variations due to fiber mixture evolution.

With the intention to better understand paper resistance and properties, the were the following tests on the laboratory sheets: Thickness and medium thickness ( $\mu\text{m}$ ) and grammage ( $\text{g}/\text{m}^2$ ) according to UNE-EN ISO 536:2012 [30]; specific volume ( $\text{cm}^3/\text{g}$ ) and specific density ( $\text{g}/\text{cm}^3$ ) According to standards in UNE-EN ISO 534:2012 [31]; Air resistance was calculated with a Gurley test apparatus, expressed in second (s), and air permeability according to Gurley ( $\mu\text{m}/(\text{Pa.s})$ ) according to standard UNE-EN ISO 5636-5:2015 [32]. All tests were performed in triplicates, following related standards.

**Figure 4.**

*Laboratory sheets were performed with different pulp proportions. Correspondence, from left to right: 100% of *Phytolacca* fibers, 90% of *Phytolacca* fibers, and 10% of commercial pulp; 70% - 30%; 50% - 50%; 30% - 70%; 10% - 90%; and 100% of commercial pulp.*

### 2.3.2 Grammage property and calculation process

The grammage is known as one of the most representative properties of paper when referring to the creative industry. This affects not only to the paper utilization but to its resistance and durability. The grammage is defined as the property that measures the mass per unit area of a sheet [30]. For its determination, each sample must be weighed on a calibrated scale, obtaining an average value that will allow us to calculate the final grammage. Due to the specific test conditions we are performing, the hand sheet diameter is constant at 160 mm, with a radius of 80 mm, obtaining an area of 0.02010 m<sup>2</sup>. These data are relevant as the formula for calculating the grammage depends on paper diameter and surface, calculated in 49.72 constant for 160 mm diameters.

Then, grammage is calculated as follows:

$$G = W \times S \quad (2)$$

where

$G$  is the grammage expressed in (g/m<sup>2</sup>)

$W$  is the weight expressed in g

$S$  is the surface, equivalent to 49.74 m<sup>2</sup>

### 2.3.3 Thickness and medium thickness properties and calculation process

The thickness of a sheet is defined as the distance between two surfaces of the material [30]. Usually expressed in micrometers (μm), to calculate this, it is necessary to use a specific machine whose accuracy allows for specific slight thickness variations. Positioned between two surfaces, the paper sheet is tight in at least five different points. This matrix of measurement points has to be distributed over the surface of the hand sheet to evaluate the complete material. After the calculation of the average, we can observe the thickness of the paper, which will affect further specific volume and density properties.

### 2.3.4 Specific volume and density properties and calculation process

The apparent specific volume of the sheet is the volume per unit mass, expressed in cubic centimeters per gram, and is calculated from the thickness and grammage of a single sheet as indicated [31]. Meanwhile, the apparent density is the mass per unit volume, calculated from the thickness of a single sheet or in relation to the apparent specific volume of a single sheet [31].

To calculate the specific volume expressed in cm<sup>3</sup>/g, it is as follows:

$$V_s = \frac{T_s}{G} \quad (3)$$

where

$V_s$  is specific volume

$T_s$  is specific thickness, calculated from the average of a single handsheet.

$G$  is the grammage of the laboratory sheet.

Furthermore, the specific density, expressed in g/cm<sup>3</sup>, as the inversion of specific volume, should be calculated as follows:

$$Ds = \frac{G}{Ts} \quad (4)$$

where

$Ds$  is specific density

$Ts$  is specific thickness, calculated from the average of a single handsheet.

$G$  is the grammage of the laboratory sheet.

### *2.3.5 Gurley air resistance and permeance property and calculation process*

Air resistance is the time required for a specific volume of air to pass through a unit area under a unit pressure. It should be expressed as “air resistance Gurley” and measured in seconds. For its calculation, the air is compressed by the weight of a vertical cylinder floating in a liquid using the Gurley apparatus [32]. The time it takes for a given volume of air to pass through the sample indicates the Gurley air resistance in s.

On the other hand, air permeance is the average airflow rate passing through a unit area under a specific pressure difference, expressed in (μm/(Pa.s)) [32]. For its calculation it is needed to apply the following equation:

$$Pe = \frac{135.3}{t} \quad (5)$$

where

$Pe$  is the air permeance

$t$  is the time

The equation is based on an average pressure difference of 1.22 Kpa, a test area of 642 mm<sup>2</sup> and a volume of 100 ml of air passing through the sample, measured at ambient pressure.

## **2.4 Chemical composition and optical properties analysis**

After calculating physic-mechanical properties, the chemical composition and evolution of handsheets were analyzed when developing the specified blends.

The chemical composition of performed tests was analyzed using SEM visualization at the CACTI (Help Center for Scientific and Technological Research) of the University of Vigo. Electron Microscope JEOL6100 was used in order to observe surface images of the internal structure of samples and chemical composition evaluation through variations in pulp concentrations. Images of optical microscope Nikon SZM1500 were studied to better appreciate texture and color properties of papers.

As it is possible to observe in **Figure 5**, the main composition of commercial pulp from *Eucalyptus globulus* made paper is based on carbon, oxygen, magnesium, silicon, calcium, sodium, alum, and phosphorus [22]. On the contrary, 100% *Phytolacca americana* fibers made laboratory paper demonstrated a composition based on carbon, oxygen, calcium, phosphorus and silicon (**Figure 6**) [22].

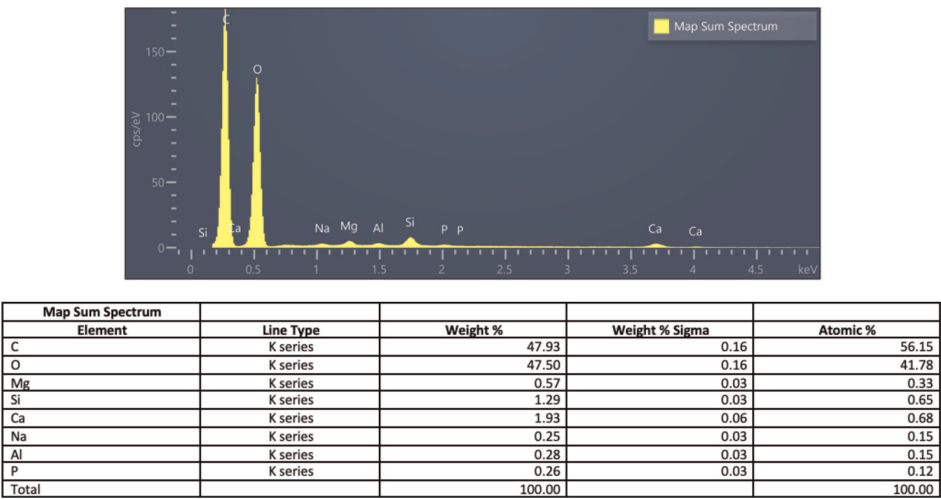


**Figure 5.**  
*Spectrum of chemical composition of 100% commercial bleached Eucalyptus globulus pulp, laboratory paper.*

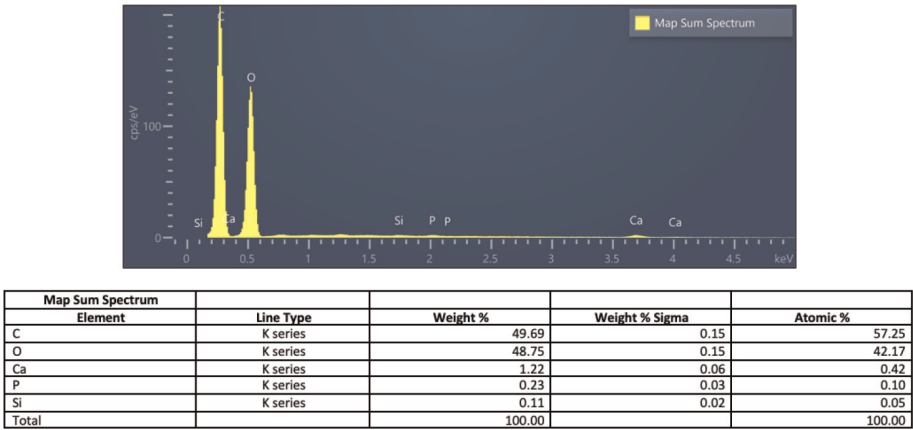


**Figure 6.**  
*Spectrum of chemical composition of 100% Phytolacca americana fiber's laboratory paper.*

In **Figure 7**, we observe how the internal structure of both papers present similarities, composed of long and thin fibers that have been measured with Image J software. Fibers presented in paper made of 100% *Phytolacca americana* pulp, demonstrated an average thickness of 8.67  $\mu\text{m}$ , with thinner fibers of 1.969  $\mu\text{m}$  and thicker one's of 18.89  $\mu\text{m}$ . The fiber length is around 200.49  $\mu\text{m}$ . In comparison, fibers that form commercial pulp handsheets are an average of 10.56  $\mu\text{m}$  thick, presenting more constant and stable fibers balancing between 5.099 and 13.99  $\mu\text{m}$ . Besides its thickness constancy, fibers are longer than 727.23  $\mu\text{m}$ , being intertwined, forming a homogeneous surface structure.

In **Figure 8**, it is possible to appreciate, from a lateral point of view, how papers of 100% *Phytolacca americana* (right) and 100% commercial pulp (left) are composed. As commented before, internal composition of *Phytolacca* papers is less ordinated than those made from commercial pulp.

Chemical analysis presented in this section allows us to observe the main composition of pure papers without mixing materials. As it will be demonstrated in the

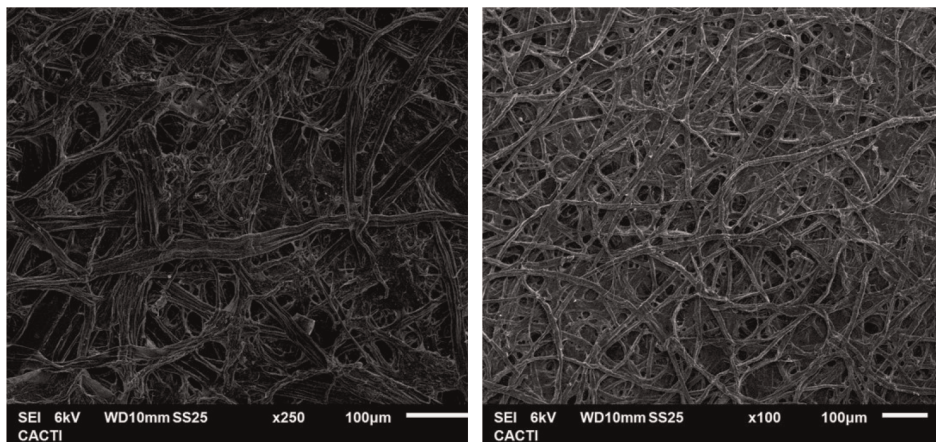


**Figure 7.**  
*SEM visualization of internal structure of 100% Phytolacca americana laboratory paper (right) and SEM visualization of internal structure of 100% commercial bleached pulp handsheet. (left).*

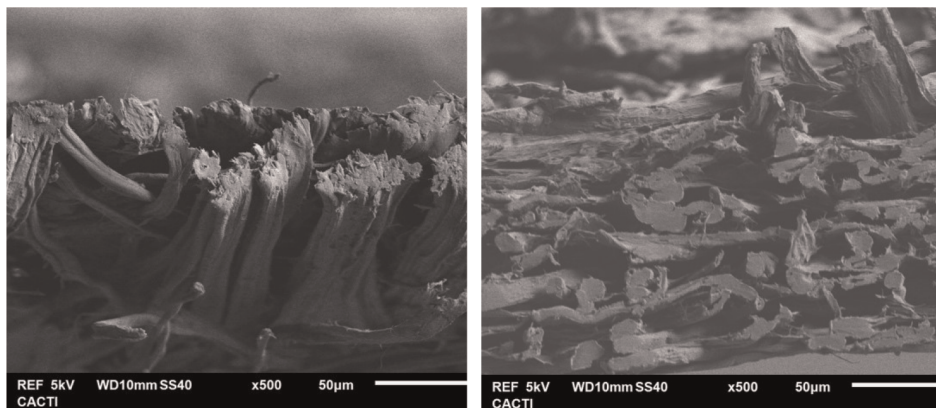


**Figure 8.**  
*Transversal section of internal structure of 100% Phytolacca americana laboratory paper (right) and Transversal section of internal structure of 100% commercial bleached pulp handsheet (left).*

following sections, these properties and compositions will be affected and suffer an evolution when blending raw matter, obtaining different results depending on the quantity.

## 2.5 Evaluation of physical, mechanical and morphological properties

Physic-mechanical and morphological characteristics were evaluated in accordance with the mentioned ISO standards, and statistical analyses were integrated into all measurements. The properties and tests were conducted and calculated in triplicate, with the data being averaged.

Mechanical properties were tested in triplicate and carried out using PFI correspondences of 1 loop = X10 in modern equipment, where 60s was deemed equivalent to 1200 rpm [22, 23]. In order to avoid misunderstandings, PFI-related data are given in seconds (s) instead of rpm.

### 3. Results

#### 3.1 Physic-mechanical properties

Experiments with different proportions of pulp blend have been named as test, corresponding test 0: 100% *Phytolacca americana* fibers pulp; 1: 90% *Phytolacca* fiber 10% commercial *Eucalyptus globulus* bleached pulp; 2: 70% *Phytolacca* fiber 30% commercial *Eucalyptus globulus* bleached pulp; 3: 50% *Phytolacca* fiber 50% commercial *Eucalyptus globulus* bleached pulp; 4: 30% *Phytolacca* fiber 70% commercial *Eucalyptus globulus* bleached pulp; 5: 10% *Phytolacca* fiber 90% commercial *Eucalyptus globulus* bleached pulp; and 6: 100% commercial *Eucalyptus globulus* bleached pulp. In **Table 2** it is possible to see the different pulp blends numbered by performed tests. This information will be referenced this way (Test 0, Test 1, Test 2...) in the following figures and tables of the document.

As it has been explained before, pulp drainability has been tested following Shopper-Riegler apparatus indications, obtaining an average degree of 36.14°SR. Higher Schopper-Riegler degrees are achieved in the extremes of proportions, being 100% *Phytolacca americana* and 90–10% mixture, in addition to 100% commercial bleached pulp with higher drainability degrees (**Figure 9**). On the contrary, even with a small

| Tests | Phytolacca matter | Eucalyptus commercial pulp |
|-------|-------------------|----------------------------|
| 0     | 100               | 0                          |
| 1     | 90                | 10                         |
| 2     | 70                | 30                         |
| 3     | 50                | 50                         |
| 4     | 30                | 70                         |
| 5     | 10                | 90                         |
| 6     | 0                 | 100                        |

**Table 2.**  
Relation of performed tests according to proportion blends between *Phytolacca americana* matter and *Eucalyptus globulus* commercial pulp.

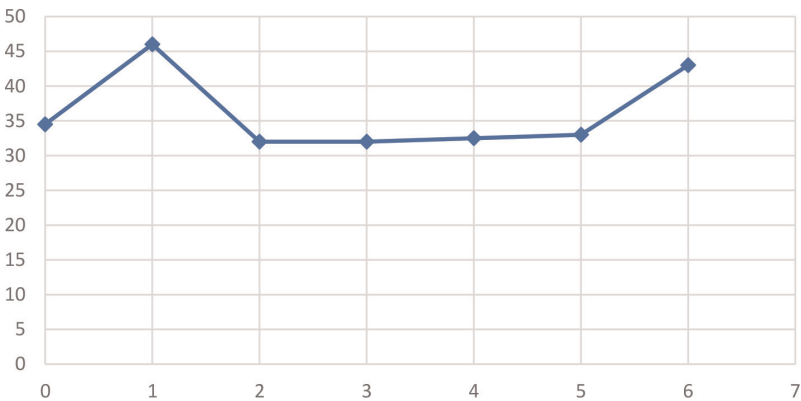


**Figure 9.**  
Drainability transformation in performed mixture test. Correspondence of Test 0 = 100% of *Phytolacca* fibers, 1 = 90% of *Phytolacca* fibers and 10% of commercial pulp; 2 = 70% - 30%; 3 = 50% - 50%; 4 = 30% - 70%; 5 = 10% - 90%, and 6 = 100% of commercial pulp.

descensus of drainability, it remains constant in intermediated blends, which indicates a homogeneous mixture and properties compensation of both raw materials.

The physical properties relation between weight and thickness is represented in **Figure 10**. It is possible to observe how thickness remains constant, with a variation of 0.0205 mm; but in relation to handsheet weight, we can observe how commercial pulp apports heavier fibers, augmenting it as we increment commercial pulp in the mixture in decrement of *Phytolacca* fibers. Lower grammages are represented by *Phytolacca americana* 100% content and 90% with an average of 23.53 and 33.49 g/m<sup>2</sup> in front of higher grammages of tests 5 and 6, which obtain an average of 64.47 and 56.7 g/m<sup>2</sup>, respectively. Medium tests with a balanced proportion of raw matter demonstrated an average of 52.39 g/m<sup>2</sup> of grammage.

In **Figure 11**, it is possible to observe grammage evolution in performed tests, deriving to a clear augment of grammage when adding commercial pulp to the mixture. Both raw matters become integrated, achieving a consistent surface. As can be observed in **Table 3**, in specific volume (cm<sup>3</sup>/g) and specific density (g/cm<sup>3</sup>) evolution, we can appreciate the same tendency, concluding higher specific density when adding commercial pulp, in decrement of specific volume (**Figure 12**).

The last physic-mechanical property studied in this research was air permeability and air resistance, measured in (μm/(Pa.s)) and seconds (s) by using the Gurley apparatus in 300 cm<sup>3</sup> measure. In **Figure 13**, it is possible to observe how air permeability decreases drastically when incrementing commercial pulp to the handsheet, contrary with air resistance, which increments softly. It is possible to suppose the relation between fiber structures of different raw matter, finding a more consistent and homogeneous surface upon commercial pulp, which increments the paper's capacity to resist air transmission. Higher air resistance times are obtained with tests 5 and 6 (9.2 and 13.15 s), corresponding with higher contents of commercial pulp. Air

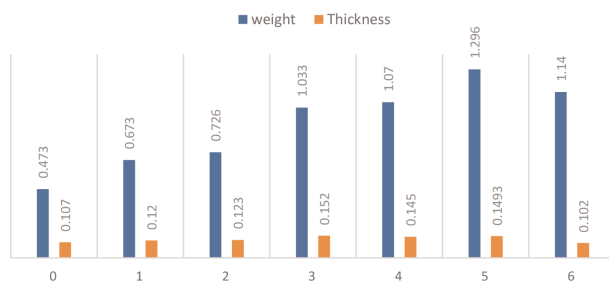


**Figure 10.**

Graphical representation of the evolution of weight (g) and thickness (mm) in performed tests.

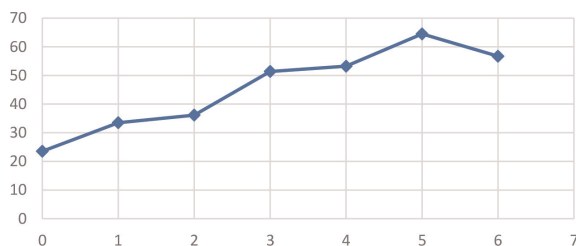


**Figure 11.**

Evolution of grammage (g/m<sup>2</sup>) in performed tests.

| Tests | Specific volume ( $\text{cm}^3/\text{g}$ ) | Specific density ( $\text{g}/\text{cm}^3$ ) |
|-------|--------------------------------------------|---------------------------------------------|
| 0     | 4.56                                       | 0.22                                        |
| 1     | 3.58                                       | 0.28                                        |
| 2     | 3.41                                       | 0.29                                        |
| 3     | 2.96                                       | 0.34                                        |
| 4     | 2.73                                       | 0.37                                        |
| 5     | 2.32                                       | 0.43                                        |
| 6     | 1.79                                       | 0.56                                        |

**Table 3.**  
 Specific volume and specific density tests results.

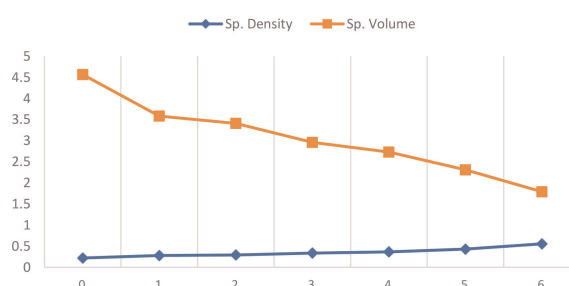


**Figure 12.**  
 Inverse relation of specific density and volume along tests.

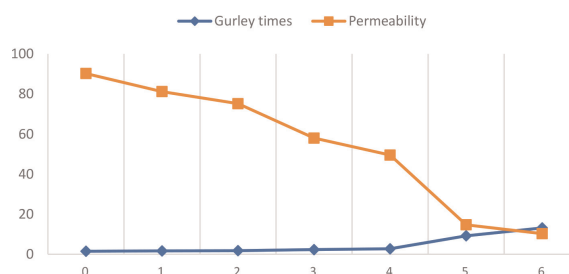


**Figure 13.**  
 Air permeability ( $\mu\text{m}/(\text{Pa}\cdot\text{s})$ ) and air resistance (s) evolution in performed tests.

permeability for these cases is the lowest obtained, being 14.7 and 10.29  $\mu\text{m}/(\text{Pa}\cdot\text{s})$ , respectively. In the case of tests 0 and 1, where *Phytolacca americana* concentration is higher, Gurley times are reduced to 1.5 and 1.66 s, while air permeability is 90.2 and 81.18  $\mu\text{m}/(\text{Pa}\cdot\text{s})$ . Medium steps, corresponding with 50% of both raw matters, present an average of 2.33 seconds and 57.98  $\mu\text{m}/(\text{Pa}\cdot\text{s})$ .

### 3.2 Internal structure integration

The internal composition of samples suffered an evolution when changing proportions of materials, but this difference is not significant when referring to surface structure due to the similitudes of fiber length and thickness. In **Figure 14**, it is possible to appreciate how main surface remains consistent, being composed of long

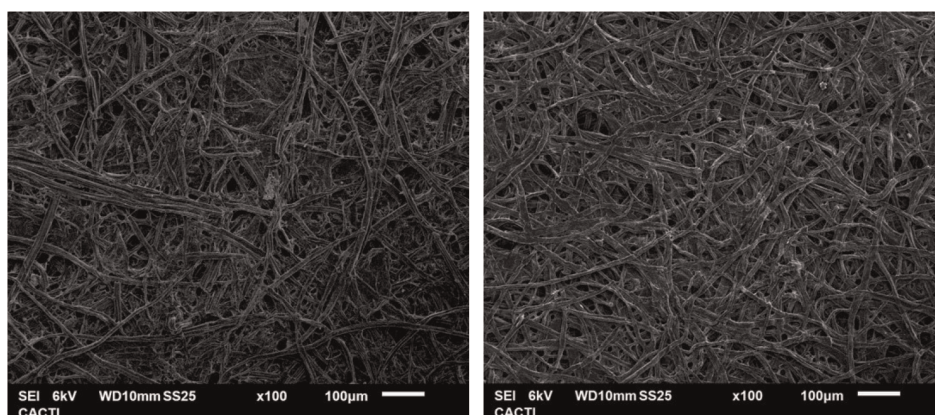


**Figure 14.**

Electron microscope images. Internal structure of handsheet performed in test 1 (90% *Phytolacca* and 10% commercial pulp) (left) and in test 5 (10% *Phytolacca* and 90% commercial pulp) (right).

fibers intertwined, creating a homogeneous surface in both cases. In **Figure 14** left, composed mainly of *Phytolacca americana* stem fibers, fibers thickness is not as consistent as in **Figure 14** right, but remains formatting a uniform surface.

In **Figure 15**, we observed how internal structure of proportions of test 2 and 4 remains similar while changing pulp blend. In **Figure 15** (left), we observe how predominant *Phytolacca*'s fibers derive in a less uniform surface, while in **Figure 15** (right), where there is a higher presence of commercial pulp, surface remains homogeneous. In case of intermediated compositions of test 3, 50% of both materials, it can be appreciated how the laboratory sheet remains formed by the same kind of fibers (**Figure 16**). In **Figure 16**, right, we observe a lateral section view of test 3 handsheet, and it is possible to observe how different fibers intertwine and form a dense structure suitable to be used for any printing, writing, or cutting ends.

In **Figure 17**, it is possible to observe how color and texture of different blends evolve when changing the proportions of materials. From left to right, *Phytolacca americana* content decreases, and commercial pulp increases. Yellow color present in

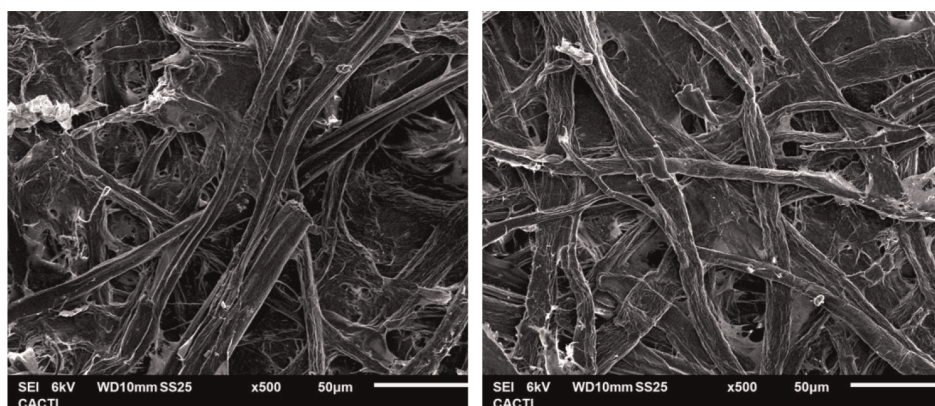


**Figure 15.**

Electron microscope image. Internal structure of handsheet formed by 70–30% of *Phytolacca* and commercial pulp (left) and surface view of internal structure of 30–70% *Phytolacca* material and commercial pulp (right).

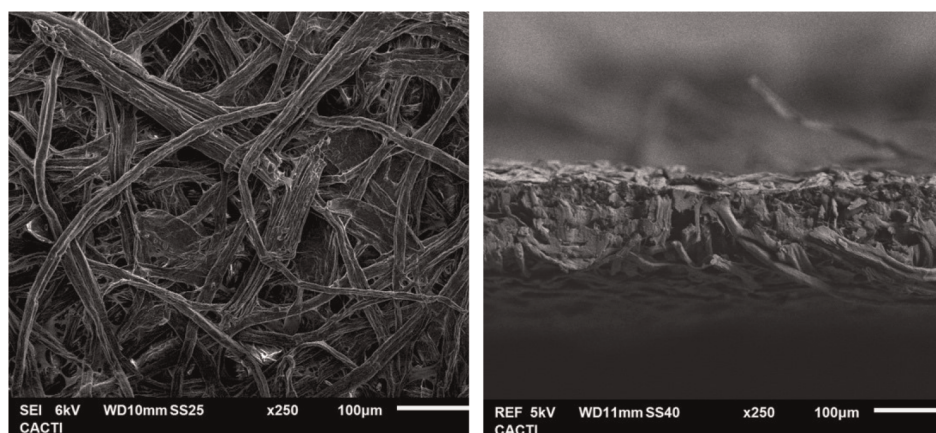


**Figure 16.** Electron microscope image. Internal structure of third test, formed by 50% of each material (left) and Lateral section view of internal structure of 50% of each material (right).

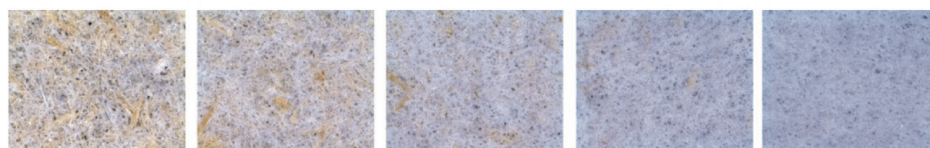


**Figure 17.** Color variations when changing proportions in laboratory sheets. Imagen obtained from optical microscope Nikon SZM1500.

unbleached pulp from *Phytolacca* gives texture and color to the laboratory sheets, adding extra value to the proposed materials.

### 3.3 Chemical composition evolution

Internal composition of samples has been studied with SEM visualization. As it has been observed, the main composition of both materials demonstrated carbon and oxygen as principal components. Differing from commercial pulp paper, *Phytolacca americana* composition presents fewer chemical compounds as it is not bleached, and this variation can be observed in **Figures 18** and **19**, representing tests 2 and 4 (70% *Phytolacca* and 30% commercial pulp – 30% *Phytolacca* and 70% commercial pulp).

As we observe in **Figures 18** and **19**, composition remains almost constant in both tests, and in **Figures 20** and **21**, we can appreciate the slight evolution when mixing both materials along different tests. The main components remain carbon and oxygen, which exchange quantities after test 2, when the major content of oxygen is present in the decrement of carbon. A higher proportion of commercial pulp derives from a higher content of oxygen. On the contrary, a higher content of *Phytolacca* material leads to a higher carbon proportion in the mixture. In **Figure 19**, we can appreciate how calcium and silicon, both secondary most present components evolve when changing the proportions of *Phytolacca* and commercial pulp. Both component's percentage tends to be 0 when augmenting commercial pulp material. Silicon disappears in test 5 when *Phytolacca* mater is just 10% of the mixture.

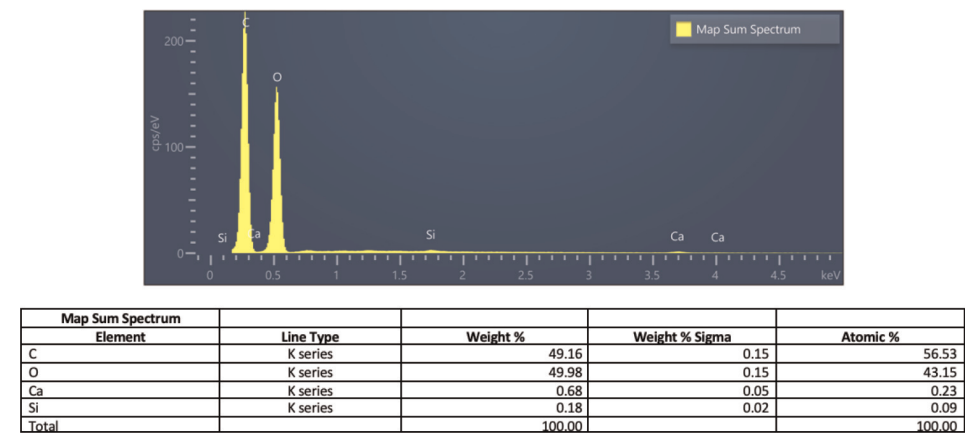


**Figure 18.**  
*Spectrum of composition in test 2.*

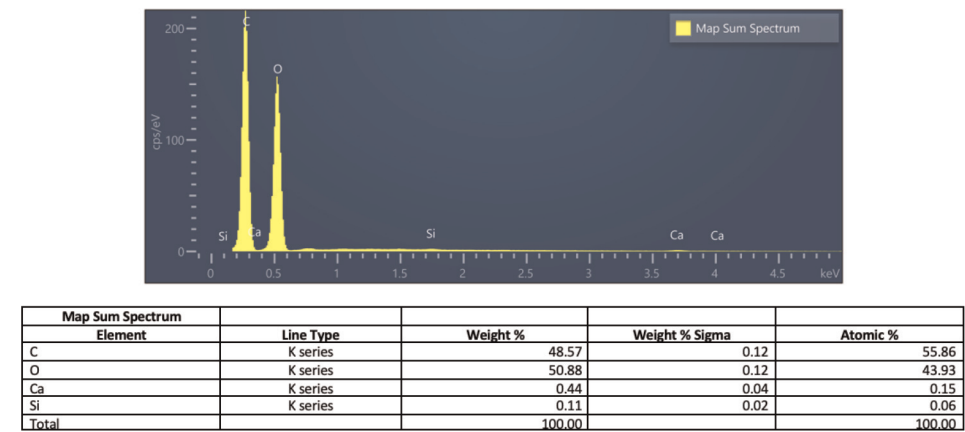


**Figure 19.**  
*Spectrum of composition in test 4.*

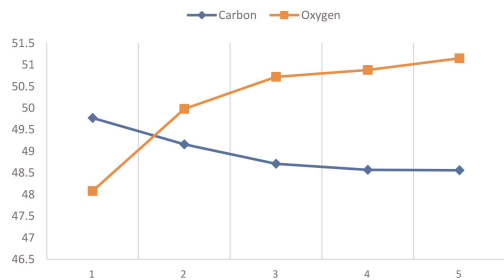


**Figure 20.**  
*Evolution of percentage of carbon and oxygen content when changing proportions of raw matter.*

As it has been demonstrated, physic-mechanical, chemical, and optical properties of developed handsheets suffered evolutions when changing material proportions. But it is relevant to mention that the difference does not reflect on the quality or resistance of material, concluding that its blend is suitable to be applied in industry.

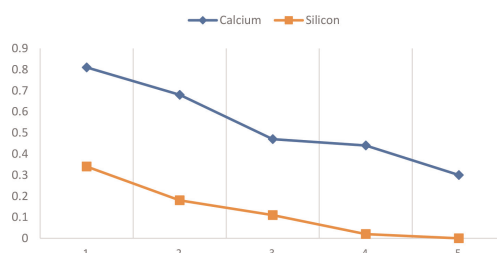


**Figure 21.**  
 Evolution of percentage of calcium and silicon content when changing proportions of raw matter.

When referring to physical properties, higher grammages are obtained with higher commercial pulp content in relation to *Phytolacca americana* material, but it remains higher with a mixture of 10–90% of *Phytolacca* and commercial pulp, than with 100% commercial pulp. Furthermore, paper-specific volume has been incremented when adding higher quantities of *Phytolacca*, and air resistance keeps constant when mixing material in the same proportions. Regarding the chemical composition of performed handsheets, there are no significant changes that will affect the biocomposability of the material; and in relation to optical properties, adding *Phytolacca americana* to commercial bleached *Eucalyptus globulus* pulp proposed value-added results, obtaining textures and colors suitable to be of interest in creative and packaging industries.

## 4. Discussion

The results obtained throughout this study demonstrate the viability of integrating the raw material from the invasive exotic species *Phytolacca americana* into paper production in varying proportions, thereby reducing the necessary amount of commercial *Eucalyptus globulus* pulp, which typically comes from monocultures. Specifically, we can observe that the drainability index of papers made with the mixture (in different intermediate proportions) remains between 30 and 35°SR (36.14°SR), while, according to [22], paper made with 100% commercial *Eucalyptus globulus* pulp with the same refining time of 120 seconds achieves values of 43°SR, indicating a slight variation in the pulp's drainage capacity. Therefore, we can say that approximately, the drainability values of the pulps are effective for commercial production. Other studies [23] show a grammage of 65.2 for paper made from bleached *Eucalyptus globulus* pulp; in this study, grammages between 20.39 and 77.26 were recorded for tests 1–5. This demonstrates that the properties of commercial paper regarding grammage remain stable in the intermediate tests, 3 and 4, where we find values of 48.08 and 54.55. On the other hand, the thickness of the samples, as previously indicated, undergoes minimal variation, remaining between 0.11 and 0.16, increasing with the addition of *Phytolacca americana* fiber. Previous studies [22, 23] demonstrate that the average thickness of commercial paper remains between 0.10 and 0.11. This property directly affects the apparent density and volume, which again experience slight variations that do not significantly affect the structural characteristics of the paper.

It is important to highlight that the slight increase in grammage, as well as the color variations contributed by the selected raw material, makes the resulting papers more valuable and particularly interesting for printing and working in creative sectors, which can benefit from the narrative potential of the material and its secondary origin.

The property with the greatest variation compared to other results obtained with commercial pulp [23] is in the Gurley air resistance and the material's air permeance. While paper made from commercial pulp presents a compact structure that prevents air passage for a longer duration, as we introduce pulp from *Phytolacca americana*, the times decrease from 3.58 to 2.51 seconds. The air permeance evolves from 81.18 to 14.70. Therefore, we can say that *Phytolacca americana* fibers affect the paper's structure, resulting in a less homogeneous constitution, which reduces its resistance to air passage.

Finally, as it was shown, the chemical composition of samples suffered an evolution when incrementing *Phytolacca americana* matter, presenting higher contents of oxygen in decrement of carbon and reducing the main composition of paper to essentials as it was avoided to use chemicals and the bleached pulp decreases in tests 3–6.

## 5. Conclusions

Conducted research on the utilization of *Phytolacca americana* fibers in combination with commercial pulp material has demonstrated significant potential for the paper industry. Value-added products demonstrate great possibilities for artistic applications and environmental communication purposes as they offer a variety of color and texturized papers along with proportions variations. The experiments undertaken have successfully optimized blending ratios, showcasing the feasibility of incorporating waste biomass from the invasive alien species *Phytolacca americana* into paper production. This approach not only promotes circular economy principles but also contributes to waste reduction initiatives, thereby mitigating the need for organic waste incineration.

The developed paper exhibits promising optical and structural properties, with varying proportions yielding diverse physical and mechanical characteristics suitable for a range of applications. Intermediated proportions of 50% of both materials demonstrate maintaining initial properties of commercial pulp paper with added values of color, texture, and fiber reutilization. The presented results confirm the feasible possibility of replacing commercial bleached pulp for proposed vegetal waste material without variation of properties, neither chemical, physic-mechanical nor optical. By harnessing waste biomass, particularly from invasive alien plant species, sustainable papermaking practices are promoted, aligning with biodiversity conservation goals and environmental stewardship principles. This innovative approach offers an eco-friendly alternative, addressing pressing environmental challenges within the paper-making sector, contributing to the possibility of reduction of wood demand for the paper industry, which will relay into monocultures reduction and local biodiversity maintenance.

As the industry faces increasing environmental pressures, embracing innovative, and sustainable methodologies is imperative for reducing ecological footprints. By promoting the adoption of such approaches, the papermaking sector can transition toward a more environmentally responsible and resource-efficient future, contributing to global efforts to combat climate change and preserve biodiversity. Simultaneously, a new paper surface with great printing and decorative ends is proposed for the creative industry, opening a huge range of possibilities in order to embrace environmental awareness and care.

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## Conflict of interest

The authors declare no conflict of interest.

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