Valorization of Seaweed: Using Brown Algae Waste in Papermaking

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Abstract

In this work, the brown alga *Dictyota dichotoma* was explored as a new reinforcing material for papermaking. Performing the typical chemical tests for cellulosic substrates on *D. dichotoma* evidenced large amounts of ethanol:benzene extractable substances (7.2%) and ashes, algae-specific results. Also, even if lipophilic compounds are removed, brown seaweed are not a primary source of fibers because it contains low proportion of cellulose. However, its elevated content of insoluble carbohydrates (51.4%) suggest there is some potential in association with conventional cellulosic pulps, as fibrous elements contribute to sheet forming and other components fill the spaces in the paper web without noteworthy loss of strength. Extraction was carried out with clean processes: hydrogen peroxide and mixtures (hydrogen peroxide-hydrochloric acid and hydrogen peroxide-sodium perborate), sodium hydroxide and sodium hypochlorite, always aiming for low reagent concentrations, in the range of 1-12%. The results show that sodium hydroxide and sodium hypochlorite were the treatments that lead to paper sheets with better structural and mechanical properties without further bleaching or refining, thus highlighting the suitability of these algae for papermaking applications.

Introduction

Depending on the meteorological conditions, tides and geography, large amounts of algae can be deposited in coastal areas. These accumulations are managed as municipal waste and often end up in landfills. Its collection is necessary because they cause environmental and health problems affecting the local tourism (Moral et al., 2019; Rajkumar et al., 2014).

Regardless of the dead biomass in beaches, since the beginning of the century there has been a notorious increase in seaweed harvest globally, from 11.4 million wet tons in 2000 to 19 million in 2010, to 28.4 million in 2014 and to 35.8 million in 2019 (FAO, 2018, 2021; Thompson et al., 2019). Cultivation for human food and cosmetics is especially common in China, Indonesia and Rep. Korea but, unless severe extractive processes are carried out, these are not safe applications for beach waste.

A frequent suggestion for the non-food valorization of seaweed is biofuel production, aiming to obtain biodiesel from the lipids and bioethanol from the saccharides (El-Sheekh et al., 2019; Patil et al., 2017). Another option, which should not be seen as competing but as complementary, is papermaking. This is a common approach to reuse agricultural waste (Aguado et al., 2018; Jiménez et al., 2008; Nagpal et al., 2020). The lack of lignin in most seaweed can make them even more appealing than field residues, as cellulose extraction becomes easier (Moral et al., 2019). However, while green algae have been successfully used as source of cellulosic fibers (Wahlström et al., 2020), the different composition of brown algae present certain challenges and, at the same time, new opportunities.

Among all the best-known types of marine algae (red, green and brown), *Phaeophyceae* (brown algae) are the most commonly found in the debris along the Mediterranean coasts (Deniaud-Bouët et al., 2014) and encompass approximately 2000 species (Thompson et al., 2019). Some studies have shown that the
area-based cultivation productivity of brown algae is higher than that of red and green algae (Lee and Lee, 2016), likely due by a higher photon absorption rate during photosynthesis (Thompson et al., 2019).

The main components in the cell walls from brown algae (up to 45% of algal dry weight) are anionic polysaccharides such as alginates and fucoidans (Kloareg and Quatrano, 1988). In some brown algae, alginate constitutes up to 60% of the total sugars (Lee and Lee, 2016). Intertidal and supratidal brown algae, including *Dictyota dichotoma* (Terauchi et al., 2012), roughly present an average weight ratio of 3:1:1 in alginates, fucoidans and cellulose, respectively (Kloareg and Quatrano, 1988). Siddhanta et al. (2011) found little variation in cellulose content among six brown algae investigated, including three different orders (*Dictyotales*, *Fucales* and *Scytosiphonales*), accounting for values around 10%.

This little amount of cellulose changes the strategy of the papermaking approach for valorization. In order to use the fibrillar cell walls with an acceptable yield and without severely damaging their structure, extraction requires particularly mild processes that, while removing most materials which hinder fiber dispersion and sheet formation, do not seek to isolate cellulose. In fact, the paper industry uses plenty of polysaccharides other than cellulose, including starch and, precisely, alginates (Bai et al., 2017). Carrageenan from red algae, which shares similarities with fucoidans, has been proven to strengthen both paper (Liu et al., 2017) and plastic films (Sudhakar et al., 2021). Hence, there are reasons to hypothesize that biomass from *Phaeophyceae*, when combined with conventional cellulosic fibers, can lead to the production of paper of good quality.

The purpose of this study is to valorize dead biomass from brown algae as a new supporting material to be added to a conventional pulp, once enriched in carbohydrates by sulfur-free and preferably mild chemical extraction methods. With this objective in mind, *D. dichotoma* was subjected to the key composition tests in papermaking. Moreover, mild extraction processes with hydrogen peroxide (alone and combined with hydrochloric acid or sodium perborate), sodium hydroxide, sodium hypochlorite and hot water were carried out. The characterization of the paper sheets fabricated with the resulting pulps was done. As far as we know, this is the first study on the production of pulp and paper including brown marine algae, and not a particular extract from them.

**Experimental**

**Harvesting and cooking**

Tidal wastes were harvested from “Playa de Costacabana” in the south of Spain (Almería). Samples were exhaustively washed with freshwater and screened in order to remove sand and other macroscopical impurities. Brown algae were selected from a mixture of marine plants and other algae, identified and dried at 40 °C during 3 days such shown in Figure 1 that schematizes the sequence of experiments.

Clean, dried brown algae were homogenized, crushed (size < 5mm), and cooked to do the extraction. Cooking was performed in a stainless steel batch reactor. Liquor to solid ratio was held at 8. Cooking liquor consisted of hydrogen peroxide, hydrogen peroxide–sodium perborate (SPB; 0.5% w/w), hydrogen
peroxide–hydrochloric acid (1% w/w), sodium hydroxide and sodium hypochlorite, all diluted with distilled water. All chemicals were supplied by Panreac AppliChem and used without further purification. Temperature and inner pressure were held constant throughout the process. The different conditions tested are summarized in Table 1. The resulting product was washed, screened, crumbled, dried (temperature: 40°C, time: 72 h) and stored at room temperature.

| Extraction agent | Dosage (w/w %) | T (ºC) | Time (min) |
|------------------|----------------|--------|------------|
| H₂O₂ (alone or with HCl or SPB) | 1 | 50 | 15 |
|                  | 3 | 65 | 60 |
|                  | 6 | 80 | 105 |
| NaOH             | 1 | 50 | 15 |
|                  | 3 | 65 | 60 |
|                  | 6 | 80 | 105 |
|                  | 9 | 80 | 105 |
|                  | 12 | 80 | 105 |
| NaClO            | 1 | 50 | 15 |
|                  | 3 | 65 | 60 |
|                  | 6 | 80 | 105 |
|                  | 9 | 80 | 105 |
|                  | 12 | 80 | 105 |

**Table 1**  
*Dictyota dichotoma* cooking conditions.

**Chemical characterization**

The pulp obtained was characterized chemically in accordance with the common TAPPI test methods for raw materials and/or pulps (TAPPI 2019). The samples for analysis were prepared according to T 264 cm-07. Solid-liquid extractions followed T 204 cm-17 for ethanol-benzene extractives and T 207 cm-08 for hot water solubility, while the ash content was determined by means of a muffle furnace in accordance with T 211 sp-11. The test for the determination of acid-insoluble (Klason) lignin was carried out with H₂SO₄ 24N (T 222 cm-15). Between 3 and 6 repetitions were carried out for each experiment and all solutions were provided by Panreac.

In the case of seaweed, as shown in previous works (Moral et al., 2019), this test targets acid-insoluble compounds that do not correspond to lignin, and thus they will be referred as Klason-positive compounds. Likewise, the results from T 203 cm-09, generally followed to estimate the content of alpha-cellulose, has to be understood here as the carbohydrate fraction which is resistant to consecutive
treatments with 17.5% and 9.45% NaOH solutions. A chlorite oxidation was carried out on a sample which had undergone water and ethanol-benzene extractions to measure the total water-insoluble carbohydrate content, since the ClO$_2$ formed in the process does not target polysaccharides (Ahlgren and Goring, 1971).

Sheet forming and testing

The conventional sheet-former method (ISO 5269-1) was used. Sheets from D. dichotoma pulps have low values of mechanical properties. For quality paper, pulp from brown algae was mixed in different proportions with an unbleached pine kraft pulp (PKP) obtained from the wood of Pinus pinaster Ait., from industrial origin. Agitation was done by hand, with a standard stirrer. Couch weights and standard plates were used to collect the handsheets. Sheets were left at 23ºC and 50% RH, while pressed by drying rings, for 24 h. The grammage of the handsheets was 60 g/m$^2$. The tensile test for the breaking length and stretch, the burst test and the tear test were performed by means of appropriate testing machines from Hounsfield, Metrotec and Messmer, respectively, and in accordance to the ISO standards 1924, 1974 and 2470 (ISO TC/6, 2011). Brightness was determined by means of a spectrophotometer from Lorentzen & Wettre, following ISO 2470 (ISO TC/6, 2011). Between 5 and 10 repetitions were carried out for mechanical properties and between 10 and 20 repetitions were performed for brightness.

Results And Discussion

Chemical characterization

The results of the characterization of Dictyota dichotoma dead biomass, not being a lignocellulosic material, have to be taken with caution. In this case, the TAPPI standards commonly used to estimate the chemical composition of plant biomass do not give out percentages of lignin, $\alpha$-cellulose and hemicellulose, but insights into the technological feasibility of using this material for papermaking. During the process, the biomass is involved in treatments with hot water, alkaline media, etc., and the end product is expected to be free from proteins and lipids. Table 2 displays the results of the tests. The amplitude of confidence intervals is four times the standard deviation.

The ethanol:benzene extractables, hot water solubles and ashes, however, are qualitatively similar to what can be expected from vascular plants, allowing for a more comprehensive comparison. Wood is known to have very low contents of lipophilic compounds, not exceeding 2.6 % in Pinus pinaster and 1.2 % in Eucalyptus globulus (Jiménez et al., 2008). The high value detected in Dictyota dichotoma (7.2 ± 1.1%) can be explained by the abundance of lipids, mainly diacylglycerol derivatives, and the presence of pigments of the algae highlighting different types of chlorophyll, fucoxanthin and beta-carotene (Ryabushko et al., 2019). In any case, similar or even larger amounts of lipophilic compounds, as found in date palm rachis (6.3% in ethanol–toluene) (Khiari et al., 2010), Arundo donax (7.3%), sorghum stalks (8%) (Jiménez et al., 2008) or Tunisian vine stems (11.3%) (Mansouri et al., 2012), have not made researchers refrain from proposing those alternative materials for papermaking.
Table 2
Chemical characterization of *Dictyota dichotoma* and comparison with other raw materials. HWS: hot water solubility. EBE: ethanol-benzene extractives. WICH: water-insoluble carbohydrate content. ARCH: alkali-resistant carbohydrate content. KLAS: acid-insoluble compounds.

| Genus       | *Dictyota* | *Ulva* | *Rhizoclonium* | *Cladophora* |
|-------------|------------|--------|----------------|--------------|
| HWS (%)     | 20.2 ± 0.6 | 33.4   | 34.6           | -            |
| EBE (%)     | 7.2 ± 1.1  | 3.8    | 9.43           | -            |
| ASH (%)     | 15.8 ± 3.1 | 19.8   | 15.9           | 2.48         |
| WICH (%)    | 51.4 ± 4.5 | 47.8   | 44.1           | 21.4         |
| ARCH (%)    | 30.6 ± 1.1 | 40.7   | -              | 17.1         |
| KLAS (%)    | 16.1 ± 0.5 | 7.9    | 3.8            | 4.64         |
| Source      | This work  | Moral *et al.* 2019 | Chao *et al.* 1999 | Mukherjee and Keshri 2019 |

A fifth of the seaweed mass, including inorganic salts and some carbohydrates, was found to be soluble in hot water (20.2 ± 0.6 %). Seemingly, inorganic salts accounted for most of that, as the ash content in *D. dichotoma* was as high as 15.8 ± 3.1 %. Sand, deposits or encrusted carbonates greatly contribute to the mineral fraction. In spite of silt being commonly removed during ash determination, in practice it represents part of the chemical composition of the harvest (Sculthorpe, 1967). Rupérez and Saura-Calixto (2011) found ashes in some Spanish seaweeds to be very abundant and variable (21-39.8%) in all the species studied.

The percentage of water-insoluble carbohydrates (51.4 ± 4.5%) is well below any cellulosic or lignocellulosic raw material, in which this fraction encompasses α-cellulose and hemicelluloses accounting for 60-80% (Jiménez *et al.*, 2008). However, it was unexpected that 30.6 ± 1.1% resisted alkaline extractions, which makes way for an easy solubilization of proteins and lipids while keeping enough material to be used as papermaking additive. Finally, the acid-insoluble content (16.1 ± 0.5%), while lower than the aromatic-rich lignocellulosic sources (26.2%) (Jiménez *et al.*, 2008), looks surprisingly high when compared to other algae (Chao *et al.*, 1999; Moral *et al.*, 2019; Mukherjee and Keshri, 2019). Taking into account the absence of structural lignin, this value can be due to certain lignin-like compounds, aromatics, alkyl derivatives and some salts.

**Carbohydrate extractions and effects on paper properties**

As can be seen in Table 3, the yield obtained after oxidation with hydrogen peroxide was lower when algae were exposed to more severe conditions. It should be remembered that, although not repeated here, a higher concentration was accompanied by a higher temperature and a longer time (Table 1). Interestingly enough, severe conditions increased breaking length, stretch, burst and tear indexes and brightness. The sheet having better characteristics was the one formed with a lower proportion of alga (25 % seaweed and 75% pine) and subjected to treatment with hydrogen peroxide and hydrochloric acid at the highest concentration (6%). Hydrogen peroxide is a powerful oxidizer that, at least without
inorganic ions, hardly targets carbohydrates but can react with aromatics, nucleic acids, lipids, or proteins (Liu et al., 2021). Hence, results indicate that the best effect is achieved by removing, by extraction, as many non-carbohydrate compounds as possible.

When H$_2$O$_2$ concentration was 3% (w/w), the presence of HCl removed more extracts and, thus, less yield, since both alkali and acid solutions help extracting proteins, and some oxidizing power should be expected from the small amounts of chlorine that are generated. This chlorine was rapidly reduced to chloride by H$_2$O$_2$ under more severe conditions (6%), in which HCl was actually a deactivator. As for sodium perborate, it has been demonstrated to be a more powerful bleaching agent than peroxide as long as high temperatures (70-80°C) are used (Pesman et al., 2014). However, possibly due to its low concentration, additions of SPB did not give out a consistent increase of brightness.

In addition, Table 3 shows that samples obtained after extracting with soda or sodium hypochlorite treatments results in paper sheets with higher quality than those obtained with hydrogen peroxide (alone or combined). In all cases, the relative standard deviation lied below 5%. As the best results were obtained at the highest concentrations of reagent, experiments with 9 and 12% of soda or sodium hypochlorite where carried out and presented in Figures 2 and 3, in order to compare both process with paper sheets formed with the greater proportion of algae (75%).
Table 3
Properties of paper sheets from *Phaeophyceae* obtained by different treatments (P: hydrogen peroxide; P-HCl: hydrogen peroxide and hydrochloric acid; P-PBS: hydrogen peroxide and sodium perborate; HW: hot water, blank). Paper sheets are formed mixing pulps of *Phaeophyceae* and *Pine pinaster* at different proportions (75:25 and 25:75). Y: Yield, TI: Tear index, ST: Stretch, BR: Burst index, BL: Breaking length and Brightness (%)

| (%) | Prop. | Y (%) | TI (Nm²/g) | ST (%) | BI (kN/g) | BL (km) | BR (%) |
|-----|-------|-------|------------|--------|-----------|---------|--------|
| P   | 1     | 75:25 | 78.7       | 4.6    | 0.24      | 0.84    | 7.8    | 45.2  |
|     | 3     |       | 70.0       | 5.8    | 0.37      | 0.92    | 7.9    | 48.4  |
|     | 6     |       | 31.8       | 7.5    | 0.63      | 2.24    | 16.5   | 50.8  |
|     | 1     | 25:75 | 78.7       | 7.6    | 0.39      | 1.16    | 7.9    | 58.0  |
|     | 3     |       | 70.0       | 7.9    | 0.58      | 1.29    | 8.1    | 58.7  |
|     | 6     |       | 31.8       | 10.4   | 0.72      | 2.48    | 23.3   | 59.2  |
| P+HCl| 1    | 75:25 | 77.5       | 6.2    | 0.36      | 0.95    | 8.2    | 46.2  |
|     | 3     |       | 50.3       | 6.6    | 0.49      | 1.08    | 7.9    | 51.5  |
|     | 6     |       | 39.4       | 8.2    | 0.68      | 2.65    | 19.6   | 51.1  |
|     | 1     | 25:75 | 77.5       | 9.4    | 0.52      | 1.09    | 7.9    | 58.0  |
|     | 3     |       | 50.3       | 10.2   | 0.66      | 1.36    | 8.2    | 58.7  |
|     | 6     |       | 39.4       | 11.7   | 0.86      | 2.99    | 30.0   | 59.3  |
| P+SPB| 1    | 75:25 | 80.9       | 4.8    | 0.20      | 0.88    | 7.6    | 50.4  |
|     | 3     |       | 76.8       | 4.8    | 0.34      | 0.96    | 7.5    | 48.8  |
|     | 6     |       | 63.1       | 8.6    | 0.55      | 2.15    | 15.9   | 50.9  |
|     | 1     | 25:75 | 80.9       | 8.0    | 0.33      | 0.96    | 7.5    | 58.1  |
|     | 3     |       | 76.8       | 8.2    | 0.47      | 1.00    | 7.9    | 57.3  |
|     | 6     |       | 63.1       | 11.5   | 0.68      | 2.65    | 20.7   | 47.8  |
| NaOH| 1     | 75:25 | 81.6       | 6.3    | 0.37      | 1.07    | 8.3    | 48.4  |
|     | 3     |       | 92.4       | 7.3    | 0.45      | 1.25    | 8.6    | 42.9  |
|     | 6     |       | 68.6       | 11.4   | 0.67      | 2.09    | 21.5   | 45.8  |
|     | 1     | 25:75 | 81.6       | 8.4    | 0.54      | 1.14    | 8.8    | 57.3  |
|     | 3     |       | 92.4       | 9.7    | 0.78      | 1.47    | 9.0    | 54.6  |
|     | 6     |       | 68.6       | 14.2   | 0.82      | 3.05    | 37.7   | 56.3  |
| (%) | Prop. | Y (%) | Tl (Nm²/g) | ST (%) | BI (kN/g) | BL (km) | BR (%) |
|-----|-----|------|-----------|-------|----------|--------|-------|
| NaClO 1 | 75:25 | 83.4 | 6.6 | 0.22 | 1.09 | 9.1 | 46.3 |
| 3 | | 76.5 | 7.5 | 0.36 | 1.22 | 9.5 | 45.6 |
| 6 | | 57.5 | 8.2 | 0.77 | 2.62 | 29.6 | 66.4 |
| 1 | 25:75 | 83.4 | 8.2 | 0.35 | 1.03 | 9.7 | 58.2 |
| 3 | | 76.5 | 9.0 | 0.50 | 1.47 | 10.2 | 57.2 |
| 6 | | 57.5 | 13.5 | 0.80 | 2.91 | 37.5 | 62.4 |

| (%) | Prop. | Y (%) | Tl (Nm²/g) | ST (%) | BI (kN/g) | BL (km) | BR (%) |
|-----|-----|------|-----------|-------|----------|--------|-------|
| HW 1 | 75:25 | 82.5 | 4.1 | 0.25 | 0.78 | 8.2 | 47.9 |
| 1 | 25:75 | 82.5 | 4.7 | 0.28 | 0.80 | 8.2 | 55.7 |

Both treatments lead to lower yields at higher reagent concentrations. However, from 9% the decrease on yield percentage is less abrupt (Figure 2a). Tear index, burst index and stretch increase at higher concentrations of caustic soda or sodium hypochlorite during pulping and no significant differences were found with both reagent (Figure 2b-e). Brightness is independent from the concentration of NaOH and increases with the concentration of NaClO, owing to the latter's capability to oxidize pigments (Figure 2f).

**Conclusions**

*Phaeophyceae* seaweed from coastal residues could not be used alone for papermaking, due to its low cellulose content and the abundance of lipophilic compounds. However, after easily extracting most proteins, lipids and pigments, the carbohydrate-rich product from brown algae constitutes a good addition to long cellulosic fibers, allowing for acceptable paper strength even when the percentage of conventional pulp was as low as 25%. Sheets formed after extractions with NaOH or NaClO presented higher quality than those obtained with hydrogen peroxide or hot water. While brightness of paper sheets was higher after NaClO treatments, hydroxide and hypochlorite extractions differed little in mechanical properties, as both produced a four-fold increase in tensile strength. These results indicate that dead biomass from these brown algae, naturally occurring along coastlines and currently needing to be treated, can be successfully reused to partially replace wood pulp in the manufacturing of non-graphical papers.

**Declarations**

**Ethics approval and consent to participate**

Not applicable. No studies involving humans and/or animals.

**Consent for publication**

Not applicable.
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Conflicts of interest/Competing interests

The authors declare that there is no conflict of interest and that they do not have competing interests.

Availability of data and material

All data are displayed in the article itself, as the mechanical testers readily presented average values.

Code availability

Not applicable.

Authors' contributions

Conceptualization: A.M. and M.B. Methodology: A.M. and J.A. Validation: M.B., R.A. and A.T. Experimentation: J.A., A.M. and M.B. Resources: A.M. Data curation: J.A., A.M., M.B and R.A. Writing—original draft preparation: R.A., M.B. and A.T. Writing—review and editing: A.M. Visualization: A.T. Supervision: A.M. and M.B. All authors have read and approve the final manuscript.

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**Figures**
Figure 1

Diagram of the experimental procedure: collection, isolation of algal biomass, analysis and preparation of handsheets. Pictures show beakers of seaweed ready to be treated (left) and handsheets from pine wood pulp and algae (right)
Figure 2

Influence of reagent concentration on processing yield (a) and or the ISO brightness (b) of paper sheets

Figure 3

Influence of reagent concentration on paper strength: tensile properties (a, b), tear resistance (c) and burst index (d)