

# The effect of wood extractives on pulp properties of maritime pine kraft pulp

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## SUMMARY

The effect of wood extraction before pulping on pulp properties, namely the unbleached pulp colour and its bleachability, was evaluated by carrying out kraft cookings after extractive removal of maritime pinewood. The colour of the pulps thus obtained was compared to a reference pulp prepared without any wood extraction. The wood chips were extracted using different non-swelling and swelling organic solvents, as well as an alkaline solution. In the case of organic solvent extractions, acetone and ethanol-toluene appeared to be the most efficient, since ensuing pulps showed an increase in the reflectance factor by up to 7.5%. For wood alkaline extraction, the results obtained showed improvements of 10% in the reflectance factor of unbleached pulp. The pulp bleachability was studied by applying a short DE bleaching sequence. The pulps from extracted woods showed increments in the reflectance factor of more than 30% compared with the reference.

## Keywords

Maritime pine, kraft pulp, extraction; organic solvents; alkaline extraction; kappa number, reflectance factor; bleachability

## INTRODUCTION

The pulp and paper production of Portugal is essentially from two sources of wood: *Eucalyptus globulus* and *Pinus pinaster* (maritime pine). The first one is a hardwood species and a source of short fibres. It gives bleached kraft pulps with good properties and it is converted to high

quality paper, whereas the second one is the principal softwood and consequently the only source of long fibres in pulp and paper industry of Portugal. It is used in the production of unbleached kraft pulps for kraft liner paper and some other special uses. The unbleached pulps from maritime pine are darker and have worse bleachability than those obtained from other common pines, which limits their use, almost exclusively, to unbleached pulp applications.

Some research has been directed towards establishing the origin of the dark colour of the pulps from this vegetal specie. One approach with this objective was to study the chemical changes in lignin structure induced by different types of pulping and different delignification degrees (1,2). The use of surfactants as cooking additives was another attempt to improve the pulp properties in terms of colour and bleachability and to understand the origin of this poor bleachability phenomena (3,4,5). The problems are usually attributed to the structure of the residual lignin and the eventual contribution of extractives was often neglected. A portion of the wood extractives is dissolved in kraft pulping liquor, but the major part of the lipophilic extractives remains in the pulp without change. In some hardwood pulps (6,7) it has been shown that these are degraded during bleaching. Since these types of extractives are similar to those found in *P. pinaster* wood (8,9), a similar behaviour during pulping and bleaching would be expected. These extractives probably have an impact on pulp colour and bleachability, as previously reported for softwood pulps (10,11). In the literature some attempts at understanding the mechanism of coloration of pulps from other wood species as a function of extractives degradation has been carried out using model compounds and mechanical pulps (12,13).

The objective of the work reported here is much more academic than industrial. In fact, our purpose is to ascertain the role that the extractives could play in the formation of dark colour and in the poor bleachability of unbleached pulps

from *P. pinaster*. For this purpose, a program composed of three sets of experiments was performed as follows:

- 1 – Kraft pulping of the original wood in order to have a reference material.
- 2 – Kraft pulping of the wood after extraction with different organic solvents. This process is directed at reaching the objective of the study and it is not proposed that this extraction might form the basis of any industrial solution. In fact, it is difficult to envisage the use of such organic solvent at industrial scale. Of those used here however, each one is suitable for the extraction of a given extractive family, so this strategy can help to discriminate the role of each family:
  - 2.1 – Extraction of wood chips with common non-swelling organic solvents, namely: diethyl ether, dichloromethane, acetone, nitromethane and a mixture of ethanol-toluene (1:2), as well as a sequenced extraction of increasing solvent polarity following the order: diethyl ether, dichloromethane, a mixture of ethanol-toluene and finally acetone.
  - 2.2 – Extraction of wood chips with swelling organic solvents, : pyridine, *N,N*-dimethylacetamide and *N,N*-dimethylformamide.
- 3 – Pre-treatment of the wood chips with alkaline solutions. As the removal of extractives by organic solvents is not economically or technically feasible in industrial processes, an alkaline extraction was tested. One of the motivations for this was the removal of extractives previously achieved with alkaline extraction in an investigation on *P. radiata* bisulphite pulps (14). The pulps resulting from the different strategies were characterised in terms of kappa number and ISO reflectance factor. The selectivity and yield of the different kraft cookings were also studied. All the results were compared with the reference. The bleachability of the most suitable pulps, in respect to reflectance factor, was

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studied. A short DE (D: ClO<sub>2</sub>; E: alkaline extraction) bleaching sequence was chosen to observe the bleaching responses of these most suitable unbleached pulps. The use of such a short bleaching sequence for bleachability studies is commonly used in research works (15-17).

It is worth noting that the pre-treatment of wood by solutions capable of extracting rosins, fatty acids, etc. has been already reported by the Springer's research team (18-20), but with a quite different aim. In fact, they studied the effectiveness of a variety of preservative molecules (such as sodium methylthiocarbamate, sodium 2,4-dinitrophenolate, sodium chlorophenolates, sodium mercaptobenzothiazole, nickel sulphate and a mixture of Na<sub>2</sub>S and Na<sub>2</sub>CO<sub>3</sub>) on preventing the deterioration of woodchips stored outdoors. These investigations showed that after a few months of storage some chemicals were effective in preventing loss of wood substance and it had no ill effects on the resultant kraft pulp quality. All treatments retarded the losses in wood.

The same authors (19) showed that storing green southern pine chips for 3 months at 65.5°C resulted in extensive chips degradation, reduced brightness, and the formation of water-soluble acids that catalysed the hydrolysis and subsequent solubilisation of hemicelluloses. When wood chips stored at this temperature were kraft pulped, pulp yields and strength properties decreased. Pre-treatment of the wood chips with alkali (synthetic green liquor and NaOH) reduced the wood degradation and pulp yields losses, but had no effect on improving strength properties.

## EXPERIMENTAL

### Material

The wood chips from *P. pinaster* used in this study were supplied by Portucel Tejo mill. Since the delignification reaction is known to depend strongly on the size of wood chips, the fraction studied was selected as homogeneous as possible and was about 20x12x2 mm. The size of these chips was relatively low because the cooking processes were carried out using mini-digesters having a capacity of 200 ml.

### Pulping experiments

The extractions with the different organic solvents were carried out in a Soxhlet apparatus during 36 hours (Table 1). In the case of the extraction with the

sequence of solvents, it was carried out for a period of 12 hours for each solvent. The pre-treatments with alkaline solutions were performed at pH=9 and are also described in Table 1.

The extracted and the non-extracted wood were cooked with the following conditions: 21% of active alkali, 30 % of sulfidity, temperature rise: 2 hours from the ambient to 170°C and 1 hour 47 minutes at 170°C. The pulps were cooked to a H-factor of 1800. At the end of the delignification reaction, the cooked chips were extensively washed and screened in a L&W laboratory strainer.

### Bleachability experiments

Bleaching experiments were carried out using a short bleaching sequence DE, consisting of an oxidation stage with chlorine dioxide followed by an alkaline extraction step using plastic bags in a shaking water bath.

The bleaching conditions were: i) D stage - ClO<sub>2</sub> charge (%) equal to 0.2 x kappa number/2.63; temperature, 70°C; pulp consistency, 10%; reaction time, 2 hours; initial pH, 2; ii) E stage - NaOH charge (%) equal to 1/2 of the active chlorine charge used in D stage; temperature, 70°C; pulp consistency, 10%; reaction time, 1 hour.

### Pulp characterisation

The kappa number and intrinsic viscosity of the pulps were determined according to standard methods: ISO 302-1981 and ISO 5351/1-1981, respectively. The pulp hand sheets were prepared according to the ISO 5269/1-1979 standard, except for those produced with the alkaline pre-treatment. In this case the hand sheets, rather than

being dried according to standard method, were immediately dried in a L&W rapid dryer for laboratory sheets. The ISO reflectance factors of the pulp hand sheets were measured using a spectrophotometer Color Touch 2 Model ISO from Technidyne Corp. following the ISO 2470-1977 standard.

## RESULTS AND DISCUSSION

Table 2 shows the data resulting from all the pulping experiments with the extracted wood. An overview of this data indicates some differences between organic and alkaline extractions, such as lower screen rejects content and higher cellulose degradation in the latter. The effect of these extractions on pulping as well as on the colour of unbleached pulp will be discussed separately.

### Pulping After Wood Extraction With Organic Solvents

In some cases, the pulps from organic solvent extracted wood showed better results, in terms of reflectance factor, compared with the values related to reference pulps (Fig. 1). These trials gave lower coloured materials with higher reflectance factor, which was the principal objective of this work. These results appeared to correlated well with the solvent characteristics - the best results were obtained with the organic solvents that gave the best extractives removal, namely those corresponding to the lipophilic part, and this agrees with results reported previously (6). The dichloromethane (E2) removes the lipophilic extractives; the acetone (E4) as well as the ethanol-toluene mixture (E3) extracted also these compounds but also some polar

**Table 1**  
Conditions of wood chips preparation

Code	Number of Experiments	Type of Extraction Procedure
R	5	none
E1	2	Diethyl ether (DE)
E2	2	Dichloromethane (DCM)
E3	2	Ethanol-Toluene (ET)
E4	2	Acetone (Ac)
E5	2	Sequenced extraction with DE, DCM, ET and Ac
E6	2	<i>N,N</i> -dimethylacetamide (DMAc)
E7	2	<i>N,N</i> -dimethylformamide (DMF)
E8	2	Pyridine (Py)
E9	2	Nitromethane (NM)
PT0	2	NaOH aqueous solution: (pH = 9), 24h, 50°C.
PT1	2	Borax buffer: (pH = 9), 24h, 25°C.
PT2	2	Borax buffer: (pH = 9), 24h, 50°C.
PT3	2	Borax buffer: (pH = 9), 60h, 25°C.
PT4	2	Borax buffer: (pH = 9), 60h, 50°C.

**Table 2**  
Data related to pulping experiments after the different pre-treatments

Experiment	Screen yield, %	Screen rejects, %	Black Liquor RAA*, g/L	Viscosity cm <sup>2</sup> /g	Kappa number	Pulp reflectance factor, %
R	41.6	6.2	5.15	1071	51.0	20.0
E1	41.6	4.9	3.80	nd*	49.0	20.1
E2	38.2	9.1	5.05	nd	47.7	21.0
E3	36.4	8.0	5.37	1049	46.8	21.4
E4	39.4	7.6	5.14	1083	46.8	21.5
E5	42.1	5.2	3.68	nd	54.2	18.5
E6	43.0	6.0	6.14	nd	58.0	18.5
E7	41.3	8.2	6.77	nd	61.1	18.5
E8	41.0	5.0	5.52	1022	48.1	20.7
PT0	40.7	5.4	4.35	865.0	44.9	22.0
PT1	43.0	3.9	3.75	1048	47.0	20.8
PT2	45.4	1.5	5.15	957.3	48.7	20.2
PT3	43.8	1.5	5.50	1043	45.4	19.3
PT4	43.8	2.5	6.35	1079	50.8	19.1

\*residual active alkali; \*not determined;

components (21,22). This mixture appears to provide the most complete removal of extractives from the wood and the pulp (22). No single solvent is capable of removing all extractive families and successive extractions are often advantageous (21), despite the results obtained with experiment E5. Thus, dichloromethane, acetone and the ethanol-toluene mixture, and especially the later two solvents, seem to remove some extractives that control the reflectance factor of the pulps. These extractives could be degraded under thermal conditions and their degradation products could be involved in the coloration of the pulps. However, it should be noticed that the kappa number of the pulps with higher reflectance factor also decreased (Table 2), which could alone explain the colour improvement. This may suggest a lessening or even the exclusion of the role played by degradation products of extractives. Nevertheless, these improved delignifications could be

ascribed, at least partially, to the removal of the extractives, which probably implies a better liquor penetration into the wood.

The removal of extractives could also contribute to the kappa number decrease because they are compounds that are oxidisable by the permanganate solution in the Kappa determination (23). However, these higher delignification levels reflect upon the screened yield, which is lower. Surprisingly, the screened rejects of these cooking experiments were higher than in the reference one (Fig. 2).

Another feature observed with these treatments concerns the selectivity of cooking process, which seems to be better in the experiments performed with acetone-extracted wood (Fig. 3). This result is in disagreement with previous investigations (11), where the extractives removal with acetone did not influence the selectivity of the cooking process. This contradiction could be attributed to the difference of the wood specie studied.

The highly polar organic solvents (E6-

E8) were expected to swell the wood chips and consequently to remove more extractives, but the results did not show this effect.

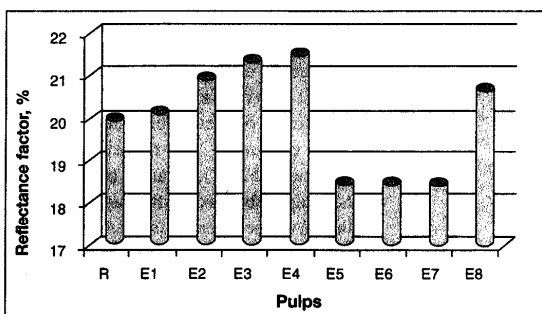
It is noteworthy that the wood extracted with nitromethane (E9), did not pulp at all even though the alkali was all consumed. (This can be inferred from the result of residual active alkali in black liquor after the cooking process, which was 0.27 g/L.)

Taking into account all the results obtained in this part of the work, we can conclude that extractions with acetone and ethanol-toluene appeared to be the most efficient, since the reflectance factor of corresponding pulps increased about 7%, when compared with the reference. Thus, these pulps were selected for further investigations concerning their bleachability.

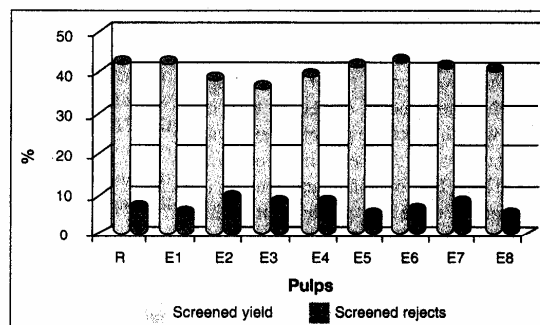
#### Pulping After an Alkaline Pre-treatment of the Wood

The alkaline treatment of unbleached pulp before bleaching has already been investigated for both decrease of lignin content (alkaline leaching) (16,23,24), and that of extractives (23,25). These treatments were carried out using more drastic conditions, in terms of pH and temperature, than those used in the present work, but at shorter reaction times. Despite these differences and the fact that the treatment was applied to wood, with less accessibility for chemical penetration, the results we obtained showed that some alkaline treatments performed during 24 hours lead to pulps with improved reflectance factor by up to 10% (Fig. 4).

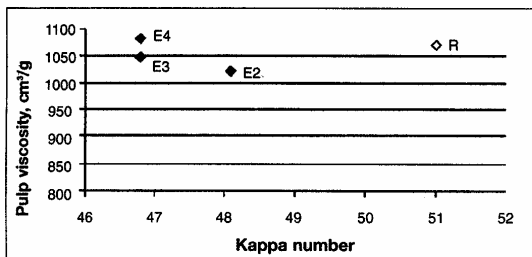
As mentioned above, this pre-treatment could remove both extractives and lignin from the wood and the observed reflectance factor improvements might only be attributable in part to extractives removal.



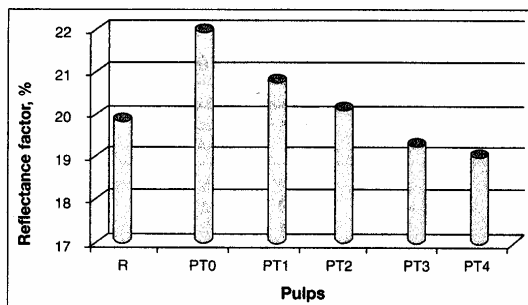
**Fig. 1** Reflectance factor of the reference pulps (R) and those obtained with organic solvents-wood extracted (E1-E8) materials.



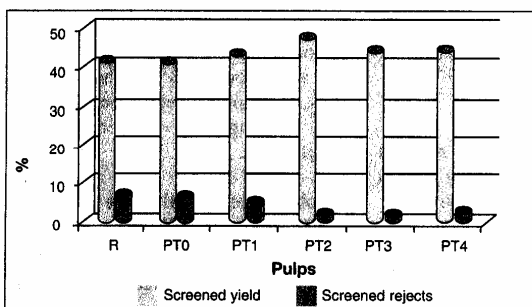
**Fig. 2** Screened yield and screened rejects of the cooking experiments described in Fig. 1 and Table 2.



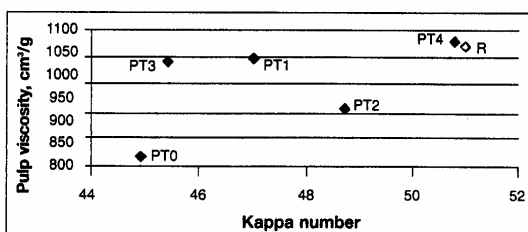
**Fig. 3** Selectivity of cooking of untreated reference pulp (R) and those obtained from dichloromethane, ethanol/toluene and acetone-extracted wood (E2-E4).



**Fig. 4** Reflectance factor of the reference pulps (R) and of those obtained with alkaline solution pre-treated wood (PT0-PT4).



**Fig. 5** Screened yield and screened rejects of the cooking experiments corresponding to the reference pulps (R) and those obtained from alkaline solution pre-treated wood (PT0-PT4).



**Fig. 6** Selectivity of cooking experiments for the reference pulps (R) and for those obtained from alkaline solution pre-treated wood (PT0-PT4).

The kappa numbers of the pulps obtained after the alkaline treatment of the wood were lower than the reference (Table 2), and this could be assigned to the extractives removal and consequent better penetration of cooking liquor, and/or to lignin removal. Anyhow, a lower screened rejects content was observed in all experiments (Fig. 5), and this was probably due to a more homogeneous wood cooking. This lower screened rejects is the principal difference compared to pulp from wood extracted with organic solvents, where the screened rejects were significantly higher than that corresponding to the cooking of the reference material. The alkaline pre-treatments gave the lower screen reject, which suggests that the removal of certain type of lignin with the alkaline pre-treatment was advantageous to the delignification process. The effect on reflectance factor is not similar, since the experiments with higher reaction times (60 hours) gave rise to pulps with lower kappa numbers when compared to the reference, but with no improvement in reflectance factor. This might be explained by a degradation of lignin structure and/or extractives and

then after the deposition of these degraded products onto the fibre surface, rendering them more difficult to be removed by the cooking process.

Another feature observed with these treatments is the small decreases in pulp viscosity in almost all pulps, which could indicate that alkaline pre-treatment of the wood, has little effect on cellulose (Fig. 6). It should also be noted that the pre-treatments with organic solvents had even less effect on viscosity (Fig. 3).

The pulps that were selected for further investigations concerning their bleachability were those resulting from the experiment PT1, even though PT0 produced higher reflectance factor. This choice was motivated by the fact that the viscosity as well as the total yield of pulps obtained in the PT0 experiment was too low. In this (PT0) experiment, the use of NaOH solution instead of a buffer, probably induced the degradation of cellulose.

#### Bleachability

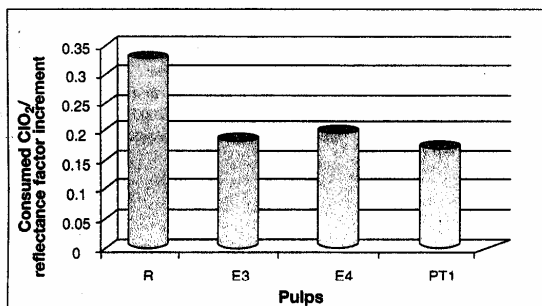
The lipophilic extractives that remain in pulp after cooking, like fatty acids, fatty acid esters and resin acids, are almost completely removed during a ECF

bleaching (6,26,27) by reaction with chlorine dioxide (28), and generally account for the the high chemical consumption (29). The results concerning the chlorine dioxide consumption, as summarised in Table 3, indicate a lower ClO<sub>2</sub> consumption, compared to the reference, for the pulps from the extracted woods. Tentatively, this result can be assigned to the removal of extractives before cooking, even though the ClO<sub>2</sub> charge was adjusted to be proportional to the ingoing kappa numbers and these are lower for the pulps E3, E4 and PT1. This lower ClO<sub>2</sub> consumption, compared to that corresponding to the bleaching of the reference pulp holds for the pre-treatment of wood by acetone and ethanol-toluene mixture, as well as with alkaline solution (pH 9).

In terms of bleachability, expressed as ClO<sub>2</sub> consumed per reflectance factor increment (Fig. 7), it can be observed that the pulps obtained with extracted wood displayed better results than those prepared from reference untreated material. This is particularly true for the pulp obtained with wood treated with an alkaline solution (PT1). This fact can be attributed not only to the initial kappa

**Table 3**  
Results of the DE bleaching of the selected pulps performed after wood extraction

Experiment	Consumed ClO <sub>2</sub> , %	Viscosity, cm <sup>2</sup> /g	Cellulose depolymerisation, %	Pulp reflectance factor, %
R	3.80	900.1	16.0	31.3
E3	3.56	914.2	12.8	40.1
E4	3.56	922.3	14.8	39.4
PT1	3.57	910.3	13.1	41.4



**Fig. 7** Bleaching response measured as the ClO<sub>2</sub> consumed per reflectance factor increment for the reference pulps (R), ethanol/toluene and acetone-extracted wood (E2 and E4) and those obtained from alkaline solution pre-treated wood (PT1).

number of the pulps but also to the absence of certain extractives and/or relating degradation products.

The viscosities of the pulps after DE bleaching sequence and respective cellulose depolymerisation (Table 3), for all the pulps studied are similar, indicating that the effects of such operations on cellulose are the same, whether the pulps were obtained from extracted wood or not, even in the case of the alkaline extraction. Moreover, it is worth noting that the viscosity and consequently the DP of cellulose after bleaching were kept at an acceptable level for papermaking application.

## CONCLUSIONS

The results obtained in this work showed clearly that the pulps obtained from wood extracted with either organic solvents or alkaline treatment were less coloured than those corresponding to the reference pulp obtained from untreated wood. Among the organic solvents, acetone and ethanol-toluene mixture appeared to be the most efficient, since the pulps obtained from the wood extracted with these solvents showed an increase in the reflectance factor by up to 7.5%. The extraction with an alkaline solution (pH 9) of the wood chips gave rise to pulps with reflectance factor improvements of 10% when compared with reference pulp.

The bleachability of these pulps, in terms of DE bleaching, was significantly better than the reference pulp, giving rise to a decrease in chlorine dioxide consumption to reach the same brightness levels.

More research is needed to understand the mechanism by which this decrease in colour takes place, as well as the origin of the pulp bleachability improvement reached with pre-extracted wood. One must determine if these improvements are due to the removal of certain extractives that during pulping, give rise to coloured degraded products or because when they are in the wood they interfere with the cooking process giving rise to pulps with higher kappa numbers. In the case of the alkaline extraction, very likely, the observed amelioration is also due to the removal of some kind of "leachable lignin" (24,30), which will negatively interfere with cooking and the bleaching processes.

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