

## HIGH CALORIFIC VALUE OF LIGNIN DERIVED FROM TURKEY OAK WOOD: COMBINED EFFECT OF STEAMING AND THERMAL TREATMENT

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**ABSTRACT:** Turkey oak (*Quercus cerris* L.) is a deciduous species characterized by high morphological variability and a widespread distribution range along South-East Europe. The wood of Turkey oak is scarcely considered as industrial lumber and is mainly used as firewood. Biomass pre-processing by heating improves feedstock consistency (mainly loss of water) and thereby improves its energetic efficiency. The main goal of this research was to evaluate how different hydro-thermal treatments effect the high calorific values and the relative ash content of lignin derived from Turkey oak wood differentially treated by combining temperature, time and steaming parameters. Sapwood and heartwood were distinguished for each treatment. Twelve different treatments were performed by using samples randomly selected. Samples were treated in a small heating unit with  $\pm 1$  °C accuracy under atmospheric pressure, according to two different heating cycles namely 120 and 180 °C. The Klason lignin content was assessed by a modified TAPPI method. High calorific value (HCV) was calculated by the fire testing technology bomb calorimeter method. Thermal treatment and the steaming processes significantly increased the HCV in examined wood samples. The strongest effect was highlighted when steaming was associated to the highest temperature. The combined effect of steaming and heating was shown to be effective in the process improvement in order to obtain both a higher content of lignin and a corresponding improvement of HCV.

Keywords: treatment, high calorific value, lignin.

### 1 INTRODUCTION

Turkey oak (*Quercus cerris* L.) is an important forest species largely present in all the South-Eastern Mediterranean countries [1]. On one side its wood has importance in economic terms due to the fact that it is used for energy purposes; on the other side, from the qualitative point of view (e.g. low dimensional stability, different technological properties between heartwood and sapwood, etc) its wood is not particularly appreciated for industrial application [2]. Heating process improves the properties of the wood (i.e. the feedstock consistency, therefore its energetic efficiency) as well as modifies the chemical composition within the wood. The high temperature causes a change and an increase in hemicellulose, cellulose, lignin, extractives content and an increase of lignin content [3]. As stated by Telmo and Lousada [4], there was a highly significant correlation between higher heating value, Klason lignin and extractive contents. Lignin is expected to play an important role as raw material for the production of bioproducts and biofuels [4]. The purpose of this research is to better understand how the steaming process and the thermal treatment can affect the High Calorific Value (HCV) of the lignin coming from the Turkey oak wood treated under different temperature, time and steaming parameters.

### 2 MATERIAL AND METHODS

The wood samples were randomly selected and twelve different treatments were performed as shown in Table I. Sapwood and heartwood were distinguished for each treatment.

**Table I:** Treatments of wood materials Ctrl Control, ST steaming treatment, H heating.

Treatment	Steaming temp. (°C)	Max pressure (bar)	Heating (2h) temp. (°C)
CTRL	-	-	-
CTRL + H 120	-	-	120
CTRL + H 180	-	-	182
ST 100	100	1	-
ST 120	120	2	-
ST 130	130	2.7	-
ST 100 + H 120	100	1	120
ST 100 + H 180	100	1	180
ST 120 + H 120	120	2	120
ST 120 + H 180	120	2	180
ST 130 + H 120	130	2.7	120
ST 130 + H 180	130	2.7	180

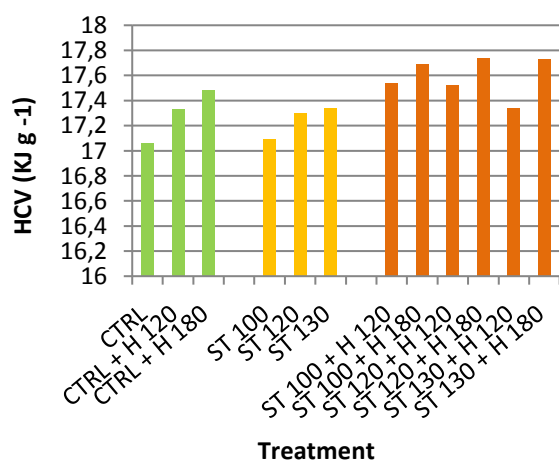
An autoclave with a maximum capacity of 23 L (Vapormatic 770/A, Asal s.r.l., Cernusco, Italy) was used in order to perform an indirect steaming process under the following temperatures and pressures parameters: 100, 120 and 130 °C at 1, 2 and 2.7 bar, respectively (Tab. 1). The instrument was sterilized by means of vertical charging which was automatic, thermo-regulated and controlled. The autoclave was equipped with a closed stainless steel basket (240 × 190 mm in diameter and height, respectively) and a microprocessor through which to program the times and temperatures (from 100 to 130 °C). The heating process was carried out putting the samples for 2 hours in a small heating unit under atmospheric pressure according to two heating cycles:

120±1 and 180±1 °C. Next the samples were cooled and weighed. Further details, such as number of specimens, procedures, etc... were reported by Todaro et al. [5].

Modified TAPPI method for the determination of Klason lignin content as the insoluble residue portion was assessed. This method required the treatment of the extractive-free material with sulfuric acid. Finally, the amount of precipitate lignin resulting from the attack of sulfuric acid on the extractive-free material was determined. These results were published in a previous study by Todaro et al. [5]. The bomb calorimeter method was used to assess the HCV of lignin. According to EN ISO 1716:2010 [6] the final results were obtained by using 3 replicated samples of lignin for each of 12 treatment, for a total of 36 measurements. The bomb calorimeter is the most common device for measuring the heat of combustion or calorific value of a material. With this apparatus a test specimen of specified mass is burned under standardised conditions. The heat of combustion determined under these conditions is calculated on the basis of the observed temperature rise while taking account of heat loss. The process of combustion is initiated inside an atmosphere of oxygen in a constant volume container, the bomb, which is a vessel built to withstand high pressures. It is immersed in a stirred water bath, and the whole device is the calorimeter vessel. The calorimeter vessel is also immersed in an outer water bath. The water in the calorimeter vessel and that of the outer bath are both monitored by a computer (Fire Testing Technology, West Sussex, RH19 4FP, UK).

### 3 RESULTS AND DISCUSSION

All treatments were compared to the control samples (CTRL). As shown in Figure 1, the thermal treatment and the steaming processes significantly increased the HCV in examined wood samples compared to control (CTRL). It is evident that the steaming treatment (ST) combined with the highest heating treatment (180°C) promote an increase of the HCV. From such results, it has been seen that the temperatures of heating treatments can strongly affect the HCV and this evidence occurred mainly in combination with steaming treatment.



**Figure 1:** HCV of lignin. Ctrl Control, ST steaming treatment, H heating.

### 4 CONCLUSIONS

The combined effect of steaming and heating treatments was found to be effective in the process improvement in order to obtain an improvement in terms of HCV. The results reported will provide information on the lignin behavior when subjected to the heating process and will be able to support future decisions on biomass management.

### 5 REFERENCES

- [1] Lavisci, P., Scalbert, A., Masson, D., & Janin, G. (1991). Quality of Turkey oak (*Quercus cerris* L.) wood. I. Soluble and insoluble proanthocyanidins. *Holzforschung-International Journal of the Biology, Chemistry, Physics and Technology of Wood*, 45(4), 291-296. <https://doi.org/10.1515/hfsg.1991.45.4.291>
- [2] Bernetti, I., Fagarazzi, C., & Romano, S. (1998, August). Biomass production as an energy source in coppices of the province of Florence, Italy. *In atti della sixth joint conference on food, agriculture and the environment*. Minneapolis.
- [3] Todaro, L., Rita, A., Cetera, P., & D'Auria, M. (2015). Thermal treatment modifies the calorific value and ash content in some wood species. *Fuel*, 140, 1-3. <https://doi.org/10.1016/j.fuel.2014.09.060>
- [4] Telmo, C., & Lousada, J. (2011). The explained variation by lignin and extractive contents on higher heating value of wood. *Biomass and bioenergy*, 35(5), 1663-1667. <https://doi.org/10.1016/j.biombioe.2010.12.038>
- [5] Todaro, L., Dichicco, P., Moretti, N., & D'Auria, M. (2013). Effect of combined steam and heat treatments on extractives and lignin in sapwood and heartwood of turkey oak (*Quercus cerris* L.) wood. *BioResources*, 8(2), 1718-1730.
- [6] UNI EN ISO 1716:2010 - Reaction to fire tests for products - Determination of the gross heat of combustion (calorific value).

### 6 ACKNOWLEDGEMENTS

The work was performed in the framework of the Suscace Project (Supporto Scientifico alla Conversione Agricola verso le Colture Energetiche).

