Correlation between the carbonization temperature and the physical parameters of porous carbons derived from *Yucca flaccida*

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**Abstract.** The purpose of the study was to develop monolithic ecological carbon materials of high porosity from the woody stems of yucca (*Yucca flaccida*). Monolithic blocks cut from the stem were carbonized in a nitrogen atmosphere, at the temperature range from 300°C to 950°C with the constant heating rate. The resultant carbon materials were characterized by dimensional changes, yield of char, elemental analysis, and various physical parameters: the true density, the bulk porosity, the longitudinal ultrasonic wave velocity and elastic anisotropy. The thermal decomposition study (TGA) was also performed. The microstructure of longitudinal and transverse sections of stems of raw and carbonized yucca were analysed by SEM. All parameters studied and the microscopic observations were discussed in relation to the pyrolysis temperature.

1. Introduction

The Nature is the biggest, the most unusual source of different kinds of environmental friendly materials which could be use in many ways of our daily life. We should benefit from the gifts of the Nature as much as it is possible. It is very important in respect to protection of natural environment.

Vascular plants are one of the best biomorphous precursors to obtain monolithic, porous carbon materials, which can be used for very wide range of applications, e.g., as activated carbons, adsorbents, catalyst supports, composites matrices etc. [1]. All cited in the literature studies involved monolithic porous carbons, which were generally derived from hardwoods or softwoods [2−6]. It would be interesting to prepare carbon materials from plants of a different structure than that of hardwoods or softwoods. Yucca (*Yucca flaccida*) is a shrub plant, belonging to a family *Agavaceae*. It is characterized by woody stem with homogeneous cross-section [7]. As a plant, it is renewable, low cost and environmental friendly material.

The purpose of this study was to develop monolithic porous carbon materials from homogenous strong stems of yucca, using highly controlled pyrolysis, and to investigate their properties vs. the carbonization temperature.

2. Experimental

Stem of yucca was carved up. Some samples of raw yucca were used for the elemental analysis, densitometry, ultrasonic and the TGA studies as well as for microscopic observations. Another part of raw yucca samples was carbonized in controlled conditions, and next the same investigations were made to determine the influence of carbonization temperature on the physical parameters of the resultant materials.

The carbonization process was performed under a gas flow of nitrogen (about 0.1 l/min) in a wide range of temperature: 300−950°C. The heating rate was constant for all final temperatures, equal to 3ºC/min. Samples were held for an hour at the final temperature.

Yield of char *Yd* was calculated from weight of raw (*w*raw) and carbonized (*w*carb) yucca sample using a relation:

\[
Yd(\%) = \left( \frac{w_{\text{carb}}}{w_{\text{raw}}} \right) \cdot 100\%
\]
The elemental analysis (carbon, hydrogen and nitrogen content) was made with the Perkin Elmer 2400 Series II CHNS/O System.

Monolithic porous carbon materials were characterized by the physical parameters such as: dimensional changes, the true density, the bulk porosity and the ultrasonic wave velocity. Reduction in dimension (shrinkage) along axial and radial directions of a sample ($RD$) caused by heat-treatment, was determined using correlation between a sample sizes measured before ($D_{\text{raw}}$) and after carbonization ($D_{\text{carb}}$) as follows:

$$RD(\%) = \left(1 - \frac{D_{\text{carb}}}{D_{\text{raw}}}\right) \cdot 100\%$$  \hspace{1cm} (2)

Both apparent and true densities were measured to determine the bulk porosity of samples. Apparent density was calculated from volume of a sample and its weight. The true density was measured using a helium gas displacement pycnometer type 1305 Micromeritics®. The bulk porosity of raw and carbonized samples was calculated using an expression:

$$P(\%) = \left(1 - \frac{\rho_{\text{app}}}{\rho_{\text{true}}}\right) \cdot 100\%$$  \hspace{1cm} (3)

where $P$ is a bulk porosity, $\rho_{\text{true}}$ and $\rho_{\text{app}}$ are true and apparent densities of a sample, respectively.

For the thermal characterization, the TGA technique was applied. Thermogravimetric studies were carried out with an analyser TGA type Q–1500, MOM Budapest, Hungary, in pure nitrogen with a flow rate of 10 l/h. Samples of about 100 mg of powdered raw and carbonized yucca were placed in a platinum crucible and they were heated from temperature 20°C to 940°C at a heating rate of 10°C/min.

The velocity of longitudinal ultrasonic wave, $v$, at a frequency of 100 kHz was measured parallel (axial direction) and perpendicularly (radial direction) to the stem axis, using an ultrasonic tester (Tester CT1, UNIPAN-ULTRASONIC, Poland). Details of an ultrasonic method are described in our previously published papers [7,8]. Ultrasonic velocities in both parallel and perpendicular to plant fibers directions were measured for yucca, before and after carbonization. Elastic anisotropy was determined as a relation $v_{\text{ax}}/v_{\text{rad}}$.

The structure of raw and carbonized yucca was observed using scanning electron microscope (SEM), Zeiss, Germany, with magnification up to 5000x.

3. Results and discussion

The thermal decomposition of yucca was investigated using thermogravimetric method. The TGA thermogram typical for yucca is shown in Figure 1. Two distinct endotherms are seen in the DTG curve (DTG is the first derivative of the TG line). The first one, at 90°C, corresponds to loss of adsorbed water. Another peak responsible mainly for decomposition of cellulose occurs at 330°C. Peak corresponding to decomposition of hemicelluloses was not observed. Probably, the temperature ranges of both cellulose and hemicelluloses decomposition couple mutually, resulting in an overlap of corresponding peaks.

![Figure 1: Thermogravimetric analysis of raw yucca](image-url)
Also peak responsible for the thermal decomposition of lignin was not found. Lignin is highly branched polymer composed of many different mers, which degradation follows at different temperatures in a wide range, i.e., of 280–500°C [2,6].

To understand changes of the yucca structure during heat treatment, the effect of carbonization temperature on the physical parameters was studied in a wide range, i.e., from 300 to 950°C. At this range of temperature, yield of char was found to diminish rapidly between 300 and 400°C (Figure 2). It is compatible with the results of the thermogravimetric studies showing distinct thermal decomposition of yucca between 270°C and 350°C (sharp minimum at the DTG curve around 330°C). Further heating causes slow changes of yield of char, although the range of temperature is wide, i.e., ΔT=550°C: the $Y_d$ value decreases from ~34% at 400°C to 28% at 950°C.

Figure 2: The plot of yield of char vs. carbonization temperature

Figure 3: Reduction in dimension in axial and radial directions (shrinkage) as a function of carbonization temperature

Figure 3 shows the dependence of the reduction of dimension measured in axial and radial directions on the temperature of carbonization. Shrinkage in both directions increases with increasing carbonization temperature and saturates at temperatures above 900°C.

The results of elemental analysis of the yucca raw and carbonized at various temperatures are presented in Table 1. Due to increasing degree of aromaticity with increasing temperature of heating, an increase of the C content, as well as decrease of the H contents were observed up to 550°C. Further heating over 550°C does not affect considerably the changes in the chemical composition in the carbonization products of yucca.

| Temperature (°C) | C (%)  | H (%)  | N (%)  |
|-----------------|--------|--------|--------|
| Raw             | 45,21  | 5,99   | 0,15   |
| 350             | 71,17  | 4,31   | 0,42   |
| 550             | 78,67  | 2,54   | 0,23   |
| 750             | 72,54  | 1,69   | 0,29   |
| 950             | 78,21  | 1,60   | 0,31   |

Table 1: Elemental analysis of raw and carbonized yucca

Figure 4 shows the plot of the true density of yucca char vs. temperature of heat-treatment. It can be seen from this figure that the true density of carbonized yucca did not reach a maximum up to 950°C. Any saturation was not observed. Probably, final reorganization of the structure of carbonized material follows at temperatures higher than 950°C.
Figure 5 presents the bulk porosity of char as a function of heat-treatment temperature. The $P$ value starts from 82% for raw yucca and reaches the maximum value of about 90% at relative low temperature, i.e., at 350°C. Carbonization of yucca at higher temperatures up to 950°C slightly increases the porosity.

![Figure 5](image)

**Figure 5:** The plot of the bulk porosity of raw and carbonized yucca vs. carbonization temperature

As it can be seen from Figure 6, the ultrasonic velocity in raw yucca rapidly decreased after heating of sample at 300°C due to the higher bulk porosity of carbonized material. Further heating up to 750°C increased the $v$ value. At this temperature, the $v_{\text{ax}}$ achieved the same value as that of the raw yucca, while the $v_{\text{rad}}$ was found to be higher. It is a result of substantial increase in rigidity of carbonized yucca structure. Heating of samples at higher temperatures produced less additional weight loss and reduction in dimensions, but formed much stiffer char than that obtained at lower temperatures. Carbonization at temperatures higher than 750°C decreased the ultrasonic velocities.

![Figure 6](image)

**Figure 6:** The plot of the ultrasonic velocity of raw and carbonized yucca vs. carbonization temperature; (O) – axial direction, (●) – radial direction

![Figure 7](image)

**Figure 7:** The plot of the elastic anisotropy of raw and carbonized yucca vs. carbonization temperature
As it was shown in Figure 7, the chars obtained at higher temperatures than \( \sim 330^\circ C \) were characterized by more isotropic properties than raw yucca.

Comparing the obtained results, it can be seen the sharp changes of the physical parameters studied at the same carbonization temperature (about \( 330^\circ C \)), characteristic for the primary thermal decomposition of yucca. The courses of the temperature dependencies of these parameters observed for higher temperatures are monotonic, approximately.

The SEM observations show that carbonization process at \( 350^\circ C \) was not finished (Figure 8(a)). Figure 8(b) reveals further changes in the structure of material carbonized at \( 750^\circ C \). It can be seen from this figure that the carbonization at higher temperatures can destroy the transverse cell walls in axial direction, which could lead to the higher surface area.

![Figure 8: Transverse section of yucca carbonized at 350°C (a) and at 750°C (b); magnification 1000x.](image)

Microscopic observations confirmed that the original cellular skeleton of yucca was retained in the porous carbon material during carbonization up to \( 950^\circ C \).

4. Conclusions

New monolithic porous carbon materials were produced from homogenous strong stems of yucca, using highly controlled pyrolysis. The optimal temperature to obtain material of the best parameters was found to be \( 750^\circ C \). Carbonized blocks of yucca were found as very light, porous, but stiff materials, which conserved the original cellular skeleton of raw plant. They are expected to be promising supports for porous, environmental friendly composites.

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