DYNAMICS OF HYDROGEN LOSS AND STRUCTURAL CHANGES IN PYROLYZING BIOMASS MATERIALS UTILIZING NEUTRON IMAGING ANALYSIS

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Introduction

Biomass is an important source for biofuel and porous carbon materials. We have been investigating material structural decay during biomass pyrolysis as well as associated hydrogen loss utilizing *in-situ* neutron radiography image analysis. At the Carbon 2018 conference in Madrid, we presented results from our first investigations that included the determination of *in-situ* hydrogen-loss dynamics inside beech wood and transmission changes in poplar up to 400 °C and neutron computed tomographies (NCTs) of wood samples. We also compared NCT with X-ray tomography on one sample that suggested that one could develop a method for obtaining relative H/C in the structure. Indeed, neutrons are sensitive to H, whereas X-rays are more sensitive to C.

In this presentation we will show new results from extended pyrolysis up to 1000 °C where the level of carbonization reached a higher level than at 400 °C. From the *in-situ* experiments, we identified different stages of hydrogen loss rate dynamics with high spatial resolution. Imaging also allows us to correlate structural dynamics of the biomass materials to the concurrent dynamics of hydrogen loss. We will also present NCTs comparing fresh and carbonized end products.

The work will include new information on results from more detailed image analysis for H/C comparing the sample introduced at the Carbon 2018 conference.

Results and Discussion

In previous work[1] we have shown that cold neutron attenuation (A) in biomass is mainly determined by hydrogen (H). During pyrolysis one notices an overall decrease in A. For this purpose, we define the natural logarithm of A, $\ln A = \ln(A)$, which is mainly proportional to H content in the biomass. We then evaluate loss of H from the expression $\ln AL = (\ln A_0 - \ln A)/\ln A_0$, where A_0 is the initial attenuation at room temperature. Pyrolysis was performed in vacuum utilizing a temperature ramp of typically 1.5 °C/min. Fig.1 shows the first and last images a movie recorded with an exposure of 30s and an average frame interval of 35s. The frames from the movie were used to calculate lnA and lnAL of each sample. Fig.1 shows that there are clear changes in

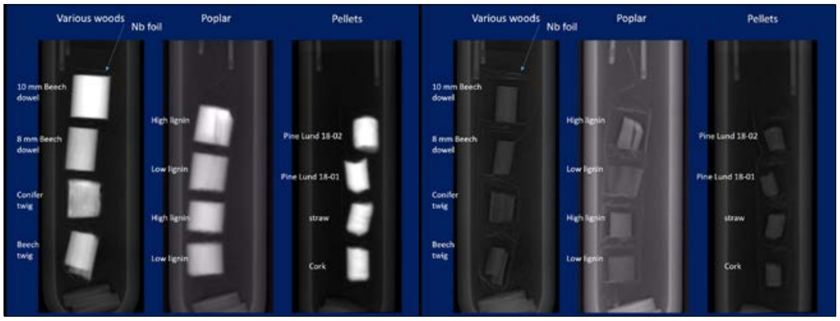

Fig. 1 lnA images of different wood and pellet samples wrapped in Nb foil inside alumina tubes before (left frame) and after (right frame) pyrolysis at up to 1000 °C.

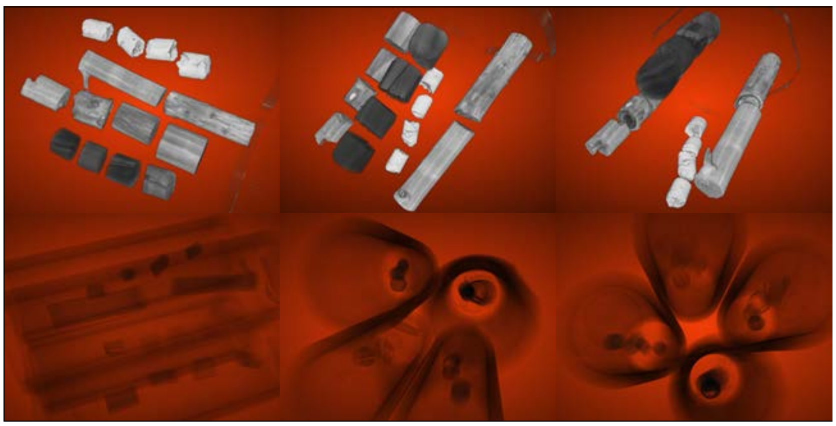

Fig. 2 NCTs on all sample tubes. Fresh samples (top row), pyrolyzed samples (bottom row).

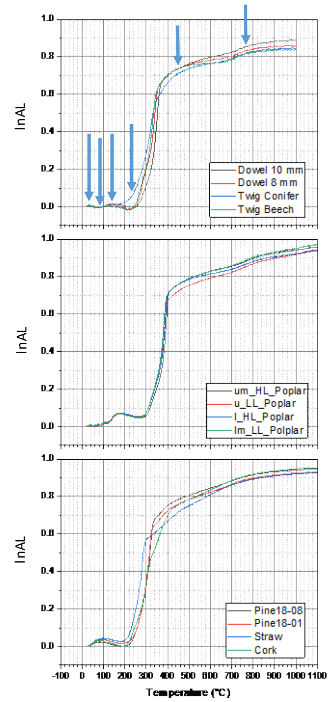

Fig. 3 Work-In-Progress on data evaluation of lnAL and H loss with temperature. Real maximum temperature was 1000 °C. 0: start; 1–5: regions of special interest.

both structural and hydrogen content between the start and end of the pyrolysis. A decreased sample brightness indicates decreased attenuation resulting from hydrogen loss in the material. Changes in shape connected to sample shrinkage are also visible. The wood structure is apparently lacking in the pellet which generally has isotropic grain orientation and shows more isotropic shrinkage. Better insights into 3D sample shrinkage can be obtained from volume views rendered from NCT imaging in Fig.2. In the presentation we will also discuss structure and hydrogen content characteristics of the 5 different regions of the pyrolysis marked by the arrows 1–5, that show different dynamics in lnAL that are related to differences in physical and chemical processes.

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