Cellulose pyrolysis investigation by thermogravimetry coupled with FTIR spectrometry

INTRODUCTION

Cellulose is a polysaccharide (Figure 1) consisting of a linear chain of several hundred to over ten thousand b(1->4) linked D-glucose units. Cellulose, located in the cytoplasmic membrane of plant cells, is the principal component and therefore the most abundant matter in the nature.
According to the Broido-Shafizadeh model [1] (Figure 2), during cellulose pyrolysis, there is a formation of an activated cellulose with a lower polymerization degree. The thermal degradation of this activated cellulose produce in one hand, volatiles and in other hand, char and gases. Proportions of each pyrolysis product depend of experimental conditions (heating rate, sample mass, gas flow…)

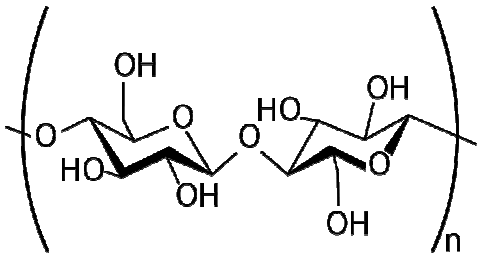

Figure 1 – Cellulose (source : kachelinfo.net)

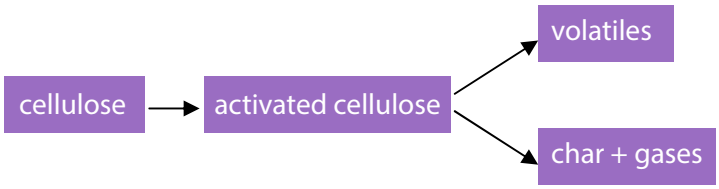

Figure 2 – Broido-Shafizadeh model [1]

EXPERIMENT

About 200mg of cellulose were introduced in a platinum container and heated from 25°C to 700°C at 10°C/min under a helium gas flow (60mL/min).
The gases released in the TGA were immediately directed towards the FTIR spectrometer. The transfer line was heated to an internal temperature of 200°C.
Each IR spectrum was obtained with a resolution of 4 cm^{-1} and the IR scanning range was from 4000 to 400 cm^{-1}.

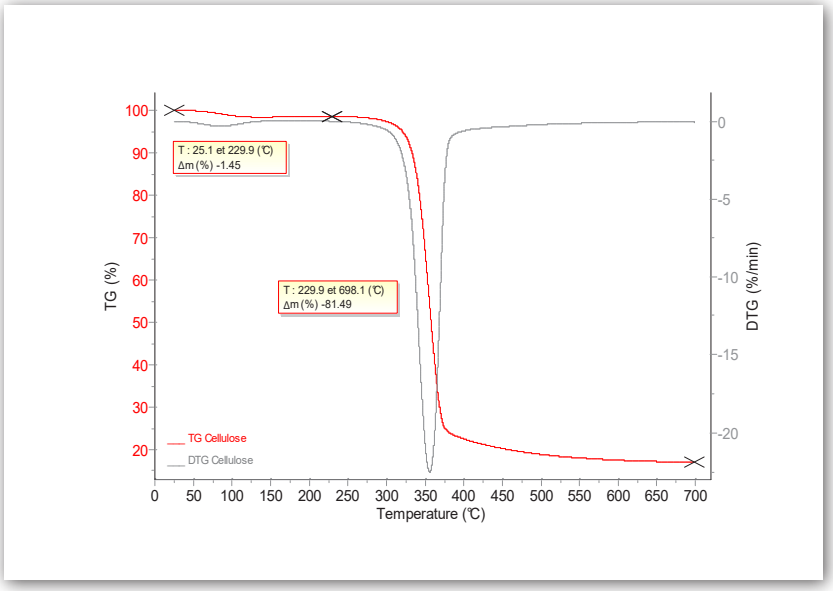

Figure 3 –Weight loss curve (%) vs. temperature of cellulose.

RESULTS AND CONCLUSION

Between 25.1°C and 230°C (Figure 3) moisture contain in cellulose is eliminated, it represent 1.45% of the initial mass. Cellulose pyrolysis is mainly carried out between 230°C and 400°C with the maximum weight loss rate of 22.57%/min attained at 356°C. This weight loss correspond to the cellulose depolymerization. When temperature is higher than 500°C, almost all cellulose is pyrolyzed with a low solid residue (19%).

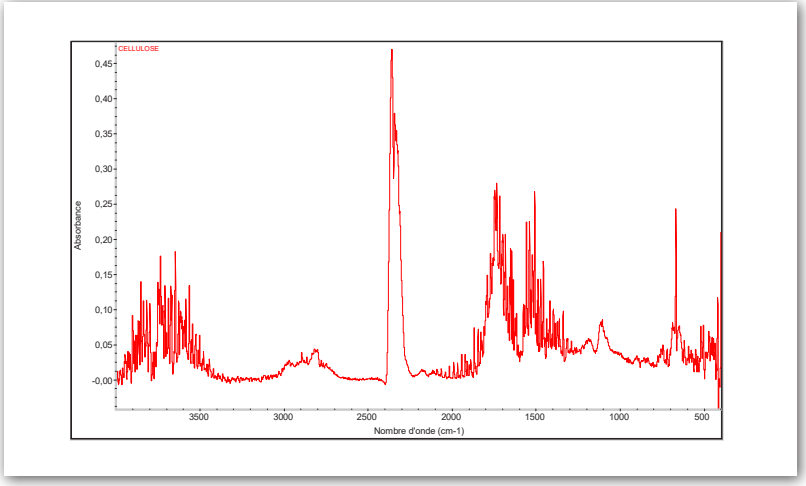

Figure 4 – FTIR spectra of gases product from cellulose pyrolysis at 364°C

Wavenb (cm^{-1})	Assignement (Bond)	Vibration	Compound	T_{max} (°C)
2143-2223	C-O	Stretching	CO	364
2020-2138	C-O	Stretching		
2237-2397	C=O	Stretching	CO_2	360
671	C=O	Bending		
3425-3991	O-H	Stretching	H_2O	364
1282-2010	O-H	In-plane rocking		
1712	C=O	Stretching	Furfural	350
754	C-H	Bending		
1651-1834	C=O	Stretching	Formaldehyde	360
2590-3132	C-H	Bending		
1680-1751	C=O	Stretching	R-CHO	364
2665-3049	C-H	Bending		
1700-1800	C=O	Stretching	R-COOH	364
1215-1301	C-O	Stretching		
2897-3300	O-H	Stretching		

Table 1 – The main products of cellulose pyrolysis characterized by FTIR and the temperature corresponding to their maximum evolution

[1] Shafizadeh F. Chemistry of pyrolysis and combustion of wood . Proc. –Int. Conf. Resid. Solid Fuels: Environ. Impacts Solutions 1982.

INSTRUMENT

THEMYS TGA/STA-EGA

EXTERNAL COUPLING CAPABILITY
designed for evolved gas analyzers (FTIR, MS, GCMS, MSFTIR, or FTIR-GCMS)

ULTRA-HIGH TEMPERATURE CAPABILITY
to 2400°C with a single furnace.

MODULAR ADAPTIONS ALLOWING
TGA only, DTA only, TG-DTA, and TMA up to 2400°C, DSC only and TG-DSC up to 1600°C all in one instrument.

HIGH ACCURACY & VERSATILITY
hang-down symmetrical beam balance, specifically designed for TGA applications