Labhydrolysis

STUDY OF SULPHITE PROCESS TO OBTAIN DISSOLVING PULP AND OTHER VALORISATION OPTIONS FROM SPENT LIQUOR

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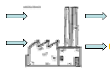
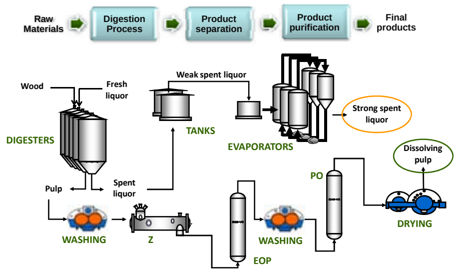
BRIGIT — SEVENTH FRAMEWORK PROGRAMME — GREEN SOURCE

CONTEXT OF THE WORK

Traditional sulphite pulp mill



Integrated Forest Biorefinery



Objective

Study of the Sulphite Process: raw materials, digestion process, kinetics and simulation of the entire process to obtain not only dissolving pulp but also other bioproducts from spent sulphite liquor.

Tasks

1.- Characterisation of lignocellulosic resources
2.- Study of the digestion process
3.- Simulation and optimisation

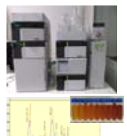
1.- CHARACTERISATION OF LIGNOCELLULOSIC RESOURCES

WOOD	STANDARD
Extractives	UNE EN ISO 14453
Cellulose	Seifert Method
Pentosans	TAPPI T222
Lignine	TAPPI T223
Holocellulose	Wise Method
Ash	TAPPI T211

PULP	STANDARD
Kappa	TAPPI 236
Viscosity	ISO 5351
α-Cellulose	TAPPI 203

LIQUOR	TECHNIQUE
Ash, water content	Furnace
Density	Picnometer
Viscosity	Viscosimeters
SO_2	TAPPI 604
Lignosulphonates	UV-VIS
Metals	AA

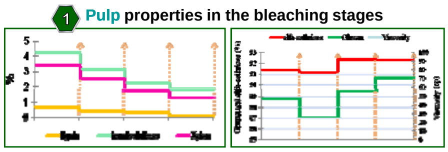
Two Step Hydrolysis



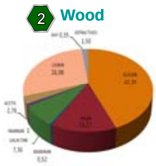
LIQUOR AND HYDROLYSATES	TECHNIQUE
Acids	HPLC-RID
Sugar compounds	HPLC-RID
Furans and Alcohols	HPLC-RID

Oligomers and Monomers

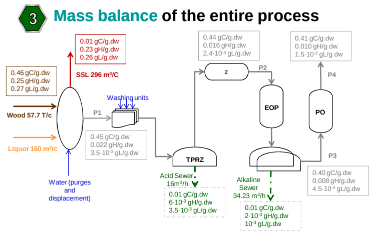
1 Pulp properties in the bleaching stages



2 Wood



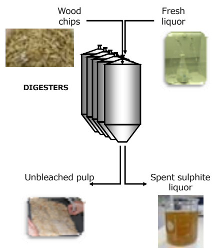
3 Mass balance of the entire process



4 Liquor valorisation

SUGAR SUBSTRATE:
Xylitol, Bioethanol, PHBs
LIGNIN:
Lignosulphonates, polymers
OTHERS
Hydrogen, fibers

2.- DIGESTION PROCESS



1 Raw material influence

WOOD SPECIE:
- *Eucalyptus globulus*
- *Eucalyptus nitens*

BASE:
- Calcium
- Magnesium

OTHERS:
- pH

	E. globulus	*E. nitens*	Ca Based	Mg Based	pH
Plantation					
Grow Resistant low temperatures and freezes Resistant against pests and diseases	orange face	green face			
Pulping					
Density Yields	green face	orange face	orange face	green face	↓
Valorisation options					
Lignosulphonates	orange face	green face	orange face	green face	↓
Fermentable sugars	green face	green face	green face	orange face	↓

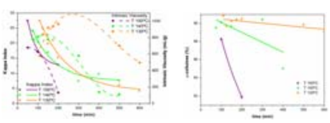
2 Operation variable influence

IMPREGNATION:
- Additives
- Time

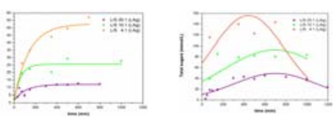
DIGESTION:
- L/S ratio
- Temperature
- Time

Experiment number	ADDITIVE (mL /L Liquor)	Impregnation (% based on first experiment)
1	A1 = 0.2	0
2	A1 = 0.1	-5.6
3	A2 = 0.2	5.3
4	A2 = 0.1	9.8
5	A3 = 0.2	7.9
6	A3 = 0.1	5.5
7	A4 = 0.2	8.7
8	A4 = 0.1	6.9

Influence on dissolving pulp parameters



Influence on valorisation options of the liquor



3.- SIMULATION AND OPTIMISATION

1 Digestion process: kinetics

Oligomers → Monomers → Decomposition products

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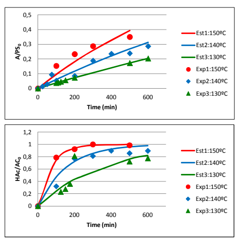
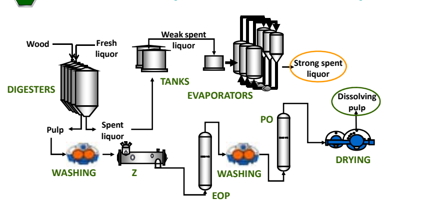


2 Simulation and optimisation of the entire process



- Sugar substrate maximisation with/without dissolving pulp: 34.88% increase with pulp. (140ºC, L/S=20/1, pH=1.5)
- **Valorisation options**: Xylitol, Bioethanol, PHBs
- Simulation of the options: future work
- Lignosulphonate maximisation: future work

3 Evaporation units: characterisation and simulation

PROPERTIES (g/L)	EHPD	HPD1	HPD2	HPD3	HPD4	HPD5
Glucose	4.17	5.45	7.45	9.69	12.86	30.88
Xylose	24.63	27.38	37.26	45.01	65.94	121.61
Galactose	3.96	6.01	6.69	7.42	10.69	18.89
Arabinose	3.05	3.2	4.97	6.41	8.19	20.17
Mannose	1.62	2.03	2.43	3.83	5.6	13.17
Acetic Acid	8.58	8.8	9.68	10.34	11.07	7.89
Methanol	1.91	1.82	1.22	2.02	2	3.56
Hydroxymethylfurfural	0.03	ND	ND	0.03	0.03	0.04
Furfural	0.14	0.16	0.08	0.06	0.02	0.19
Lignosulphonates	42.24	53.53	64.59	86.08	140.8	428.2

Maximisation of the valorisation options.
Minimisation of the inhibitors: detoxification.

4 Bleaching processes: kinetics

future work.

CONCLUSIONS AND PERSPECTIVES

- A global characterisation of all of the lignocellulosic streams in a sulphite process has been carried out giving the main losses, the recommendations for the bleaching steps and the valorisation options.
- The digestion process has been studied obtaining the best raw materials and operation variables to increase the valorisation options.
- A simulation of the entire process has been developed giving the kinetics of the digestion process and studying the evaporation units.
- A maximisation of sugar substrate in the liquor has been obtained.
- Bleaching kinetics, lignosulphonates and detoxification processes will be studied in the future.

ACKNOLEDGEMENTS

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