

# Cationisation of hemicelluloses and their use in the paper pulp

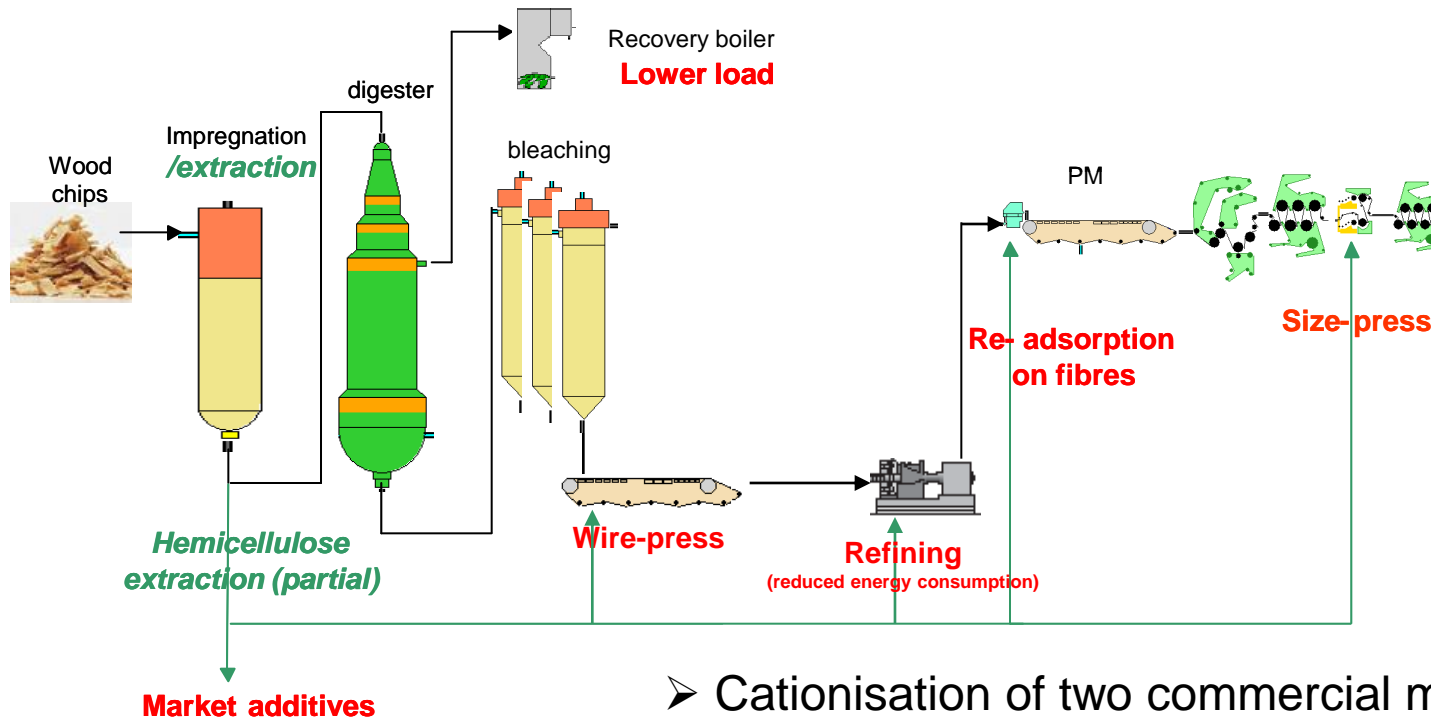
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# Motivations and objectives

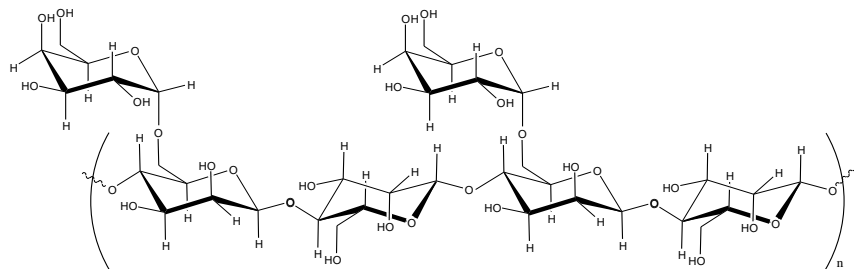
- ✓ Promotion of hemicelluloses in pulp mills
- ✓ Replacement of cationic starch in papermaking



- Cationisation of two commercial model hemicelluloses and one extracted at FCBA
- Use of cationic derivatives as dry strength aids in the paper pulp

# Hemicelluloses Cationisation

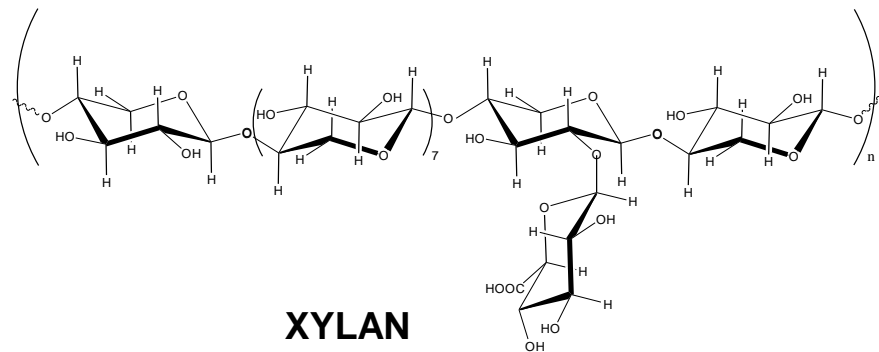
- Cationisation study on two model hemicelluloses (commercial)



**GALACTOMANNAN**  
from leguminous plant (Guar gum)

DP~6000, highly branched (**Man:Gal = 2:1**)

High viscous gel in aqueous media



**XYLAN**  
from birchwood

DP~200, low branching

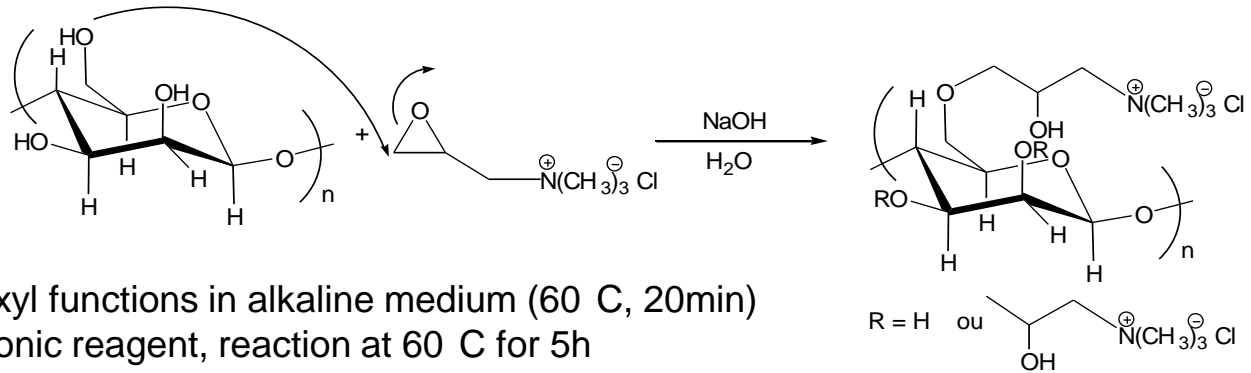
Suspension/solution in aqueous media

- Cationisation applied to a birchwood xylan extracted at FCBA

- ✓ Same structure that model xylan
- ✓ Lower DP

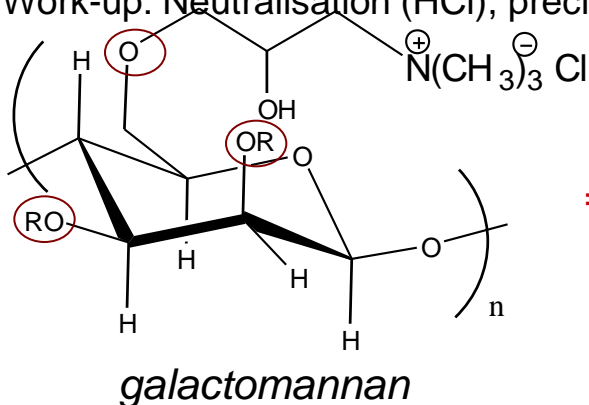
# Hemicelluloses Cationisation

## ➤ Synthesis of cationic hemicelluloses by etherification with a cationic epoxide



### ❖ Procedure

- Activation of hydroxyl functions in alkaline medium (60 C, 20min)
- Addition of the cationic reagent, reaction at 60 C for 5h
- Work-up: Neutralisation (HCl), precipitation with ethanol



Guar :  $DS_{max} = 3$   
 Xylan :  $DS_{max} = 2$

Reaction Efficiency = Substitution Degree (DS)

= Average number of grafted cationic functions by sugar unit

- DS determined by  $^1H$  NMR spectroscopy after hydrolysis in DCI/D<sub>2</sub>O
- Mass Yield of recovered hemicelluloses after reaction (taking into account the DS value)

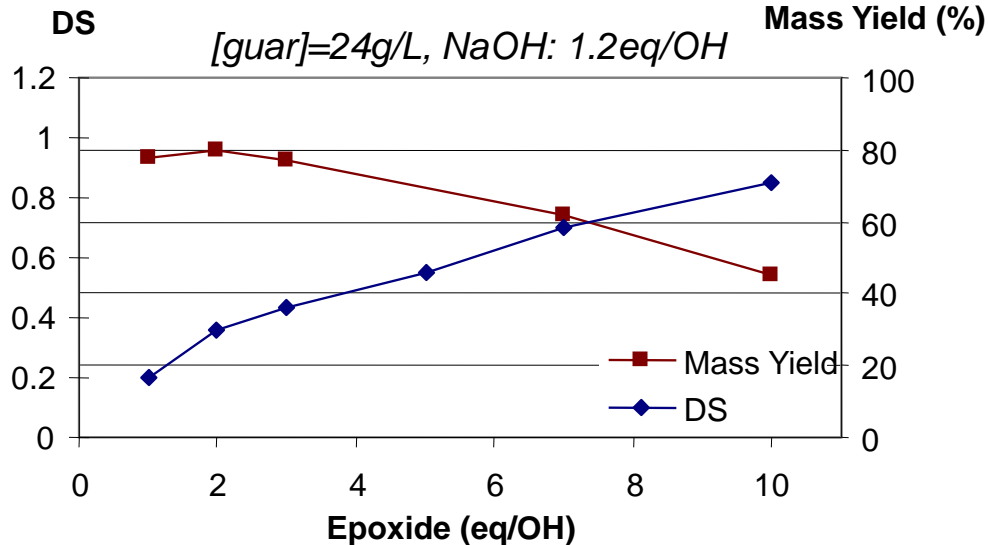
# Hemicelluloses Cationisation



## ➤ Influence of reactional parameters - Epoxide amount

### GALACTOMANNAN

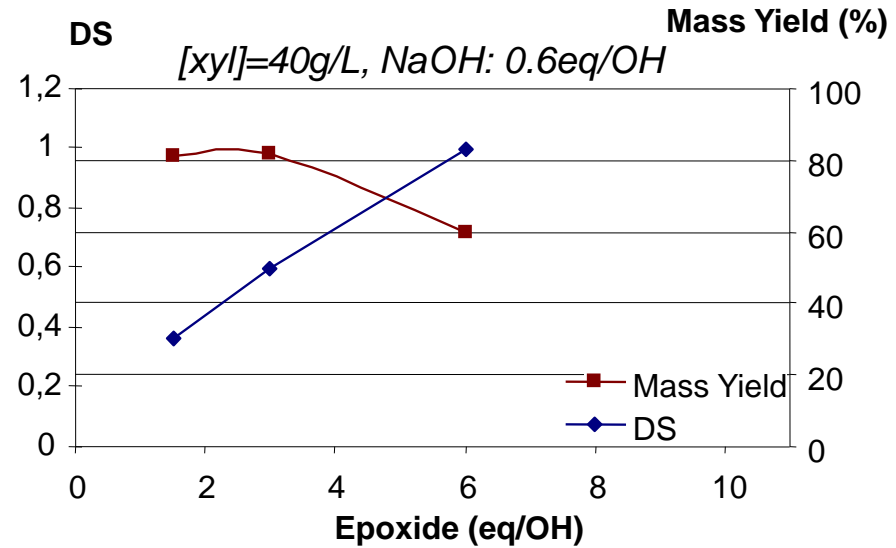
*[guar]=24g/L, NaOH: 1.2eq/OH*



➤ DS values : 0.2 to 0.8

### XYLAN

*[xyl]=40g/L, NaOH: 0.6eq/OH*



➤ DS values : 0.3 to 1

✓ In both cases: Linear increase of DS with increase of epoxide amount

✓ Decrease of mass yield with increase of DS

↪ High DS more soluble in water → less hemicelluloses recovered after precipitation in ethanol

# Hemicelluloses Cationisation



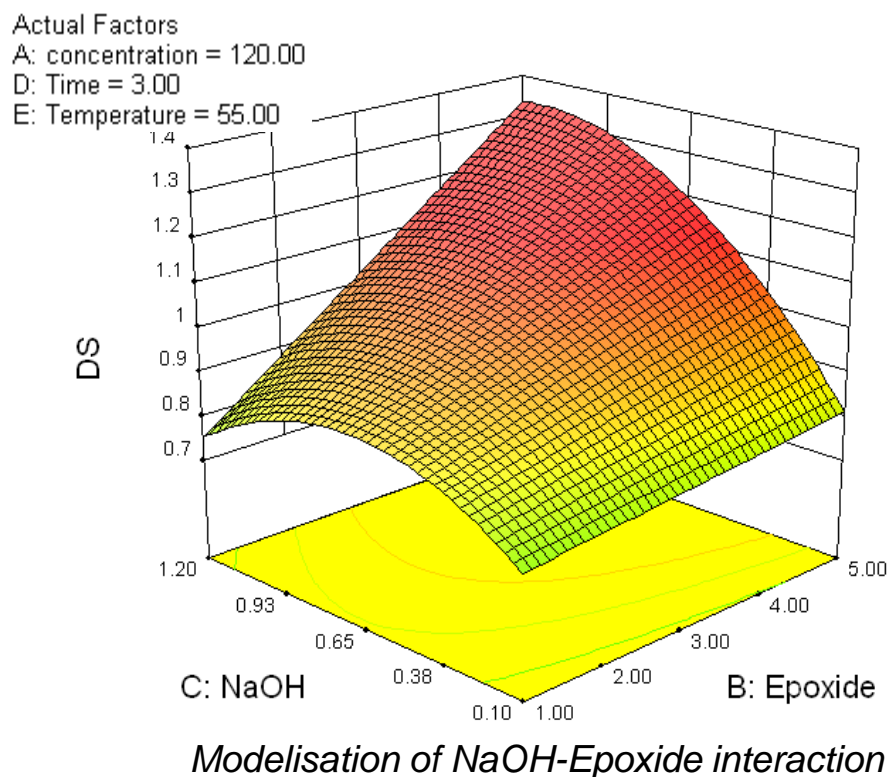
## ➤ Influence of reactional parameters - Interaction NaOH-Epoxyde

➤ **Experimental design** : Significant model ( $\text{adj-R}^2$ ) = 0.92

### ✓ 4 detected interactions

- **BC**: Epoxyde-NaOH
- **AE**: Concentration-Temperature
- **BE**: Epoxyde-Temperature
- **CE**: NaOH-Temperature

➔ **Temperature** involved in 3 interactions (Time not involved)



# Cationic derivatives as additive in paper pulp

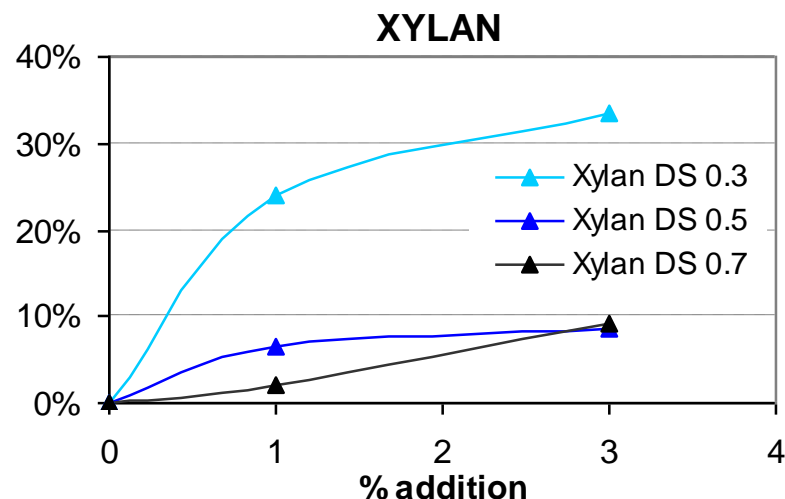
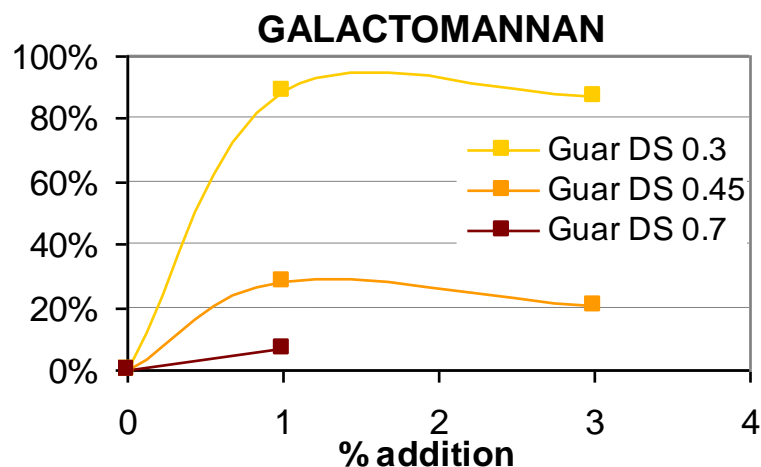


- ✓ Synthesis of cationic derivatives with DS from 0.1 to 1
  - ↪ Influence of the addition of cationic hemicelluloses in paper pulp as dry strength agent
  - ↪ Evaluation mechanical properties of paper
    - ➔ **Measure of the Burst, Tensile and Tear Indexes on handsheets**
  
- ✓ Formation of handsheets on FRET device (Retention handsheet former)
  - Use of a bleached hardwood kraft pulp (mixture of birch, beech and eucalyptus in equal proportions), refined to 34°SR
  - Hemicelluloses added in the diluted pulp (3 g/L), 1 min stirring, dewatering and sheet formation

# Cationic derivatives as additive in paper pulp

## ✓ Influence of cationic hemicelluloses with DS 0.3 to 0.7

### ▪ Variation Burst Index



➤ Improvement of paper mechanical properties by cationisation

➤ Galactomannan more efficient than Xylan (in the same range with starch)

➤ In both cases : **DS 0.3 IS OPTIMAL**

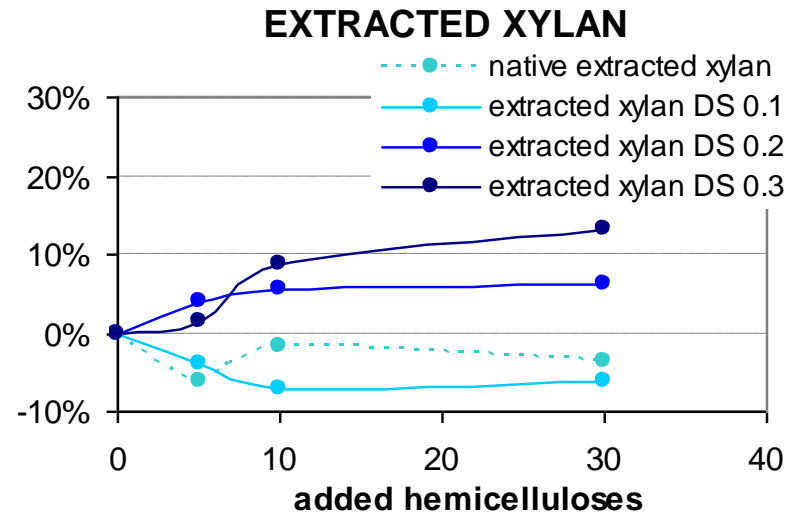
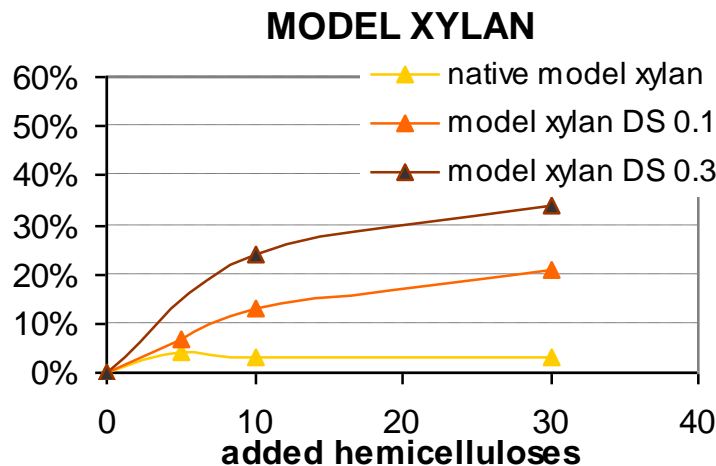
**No further improvement by increasing the DS**

**RETENTION** : 80% of added hemicelluloses retained on fibres (in all case)

# Cationic derivatives as additive in paper pulp

## ✓ Influence of LOW DS & Properties of the EXTRACTED XYLAN

### ▪ Variation Burst Index



- Extracted xylan less efficient than the model one (lower  $M_w$ )
- Loss of properties by decreasing the DS under 0.3

In the range of DS from 0.1 to 0.7  
**Optimal DS = 0.3**

- Synthesis of two families of cationic hemicelluloses (galactomannan and xylan) with various DS by modulating reactional parameters
- Identification of preponderant parameters and optimisation of conditions with two complementary approaches (Systematic study + Experimental Design)
- Study of the mechanical properties of paper after addition of cationic hemicelluloses as additive in the paper pulp
- Best results obtained for hemicelluloses with DS 0.3

**THANK YOU FOR YOUR ATTENTION**