Switching to Nano: Switchable Ionic Liquids in Biorefining provides the toolbox - from fractionation and pretreatment to catalysis and nanocellulose

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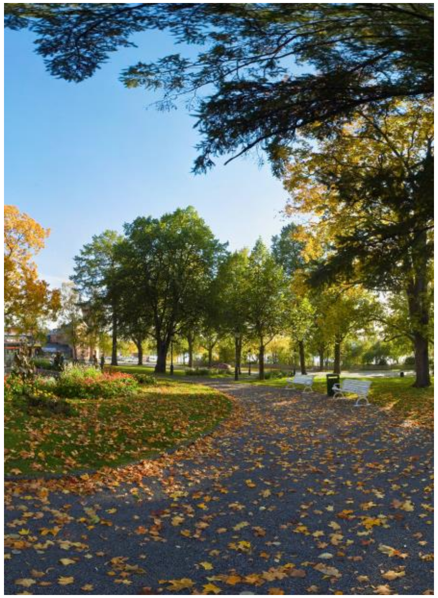
Representing the 'Switchable, Green Team' at Umeå & Åbo Akademi Universities

Åbo Akademi University

Bio4Energy



Appointed by the Swedish government to develop methods and tools for large-scale biorefinery, as one of its Strategic Research Environments.

- Research environment in northern Sweden including three universities, research institutes and more than 20 industrial or R&D companies.
- The aim is to make bioenergy and "green" chemicals out of forest-sourced raw materials and waste streams with a low environmental impact and high efficiency.
- Extensive expertise in biochemistry, plant engineering, pretreatment of feedstock, catalysis and separation methods, biomass combustion by gasification, system analysis, bioeconomy and more.

Bio4Energy – Constellation

Research joint project between:

- Umeå University UmU
- Luleå University of Technology LTU
- Swedish University of Agricultural Science SLU
- Innventia

Strategic partners: SP ETC, SP Processum

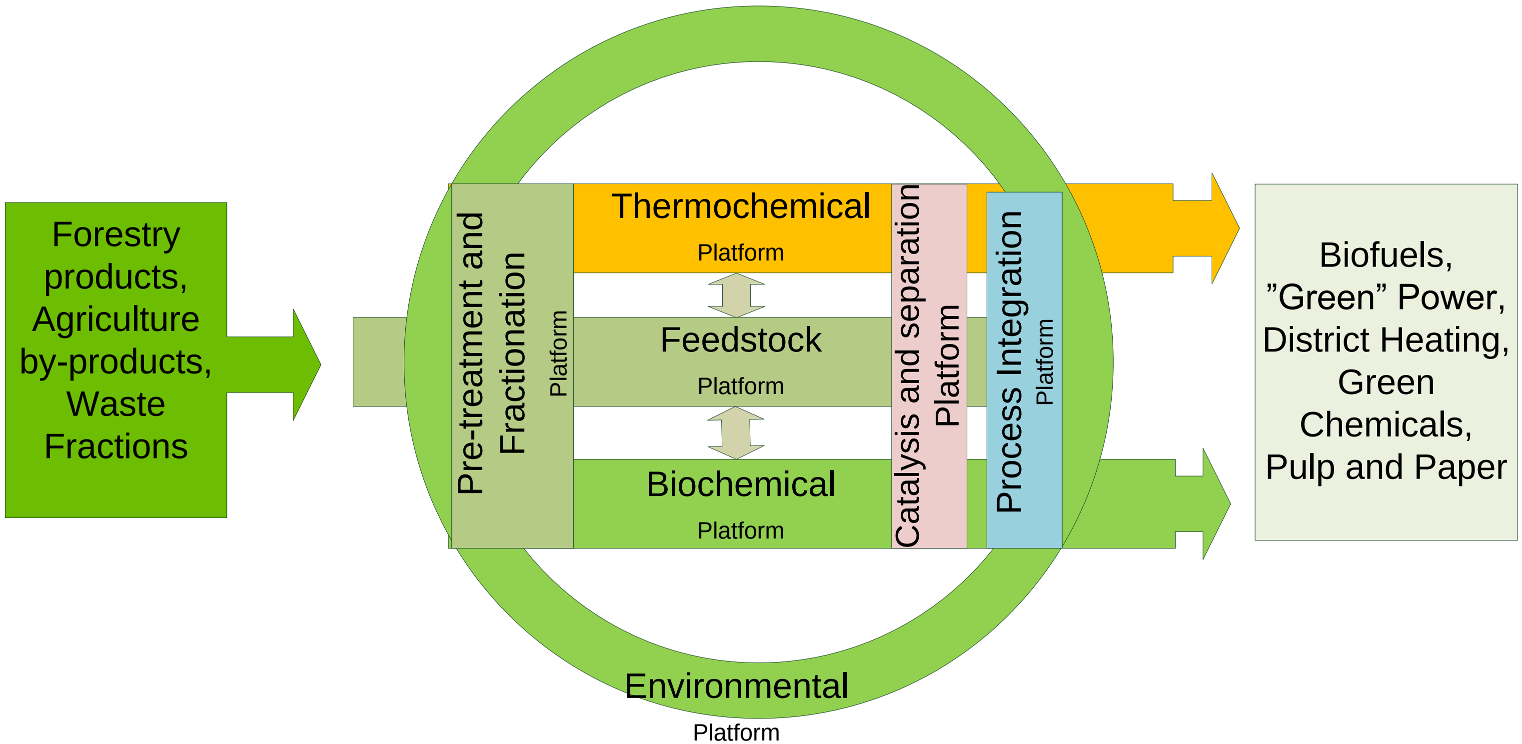
UMEÅ UNIVERSITET

LULEÅ TEKNISKA UNIVERSITET

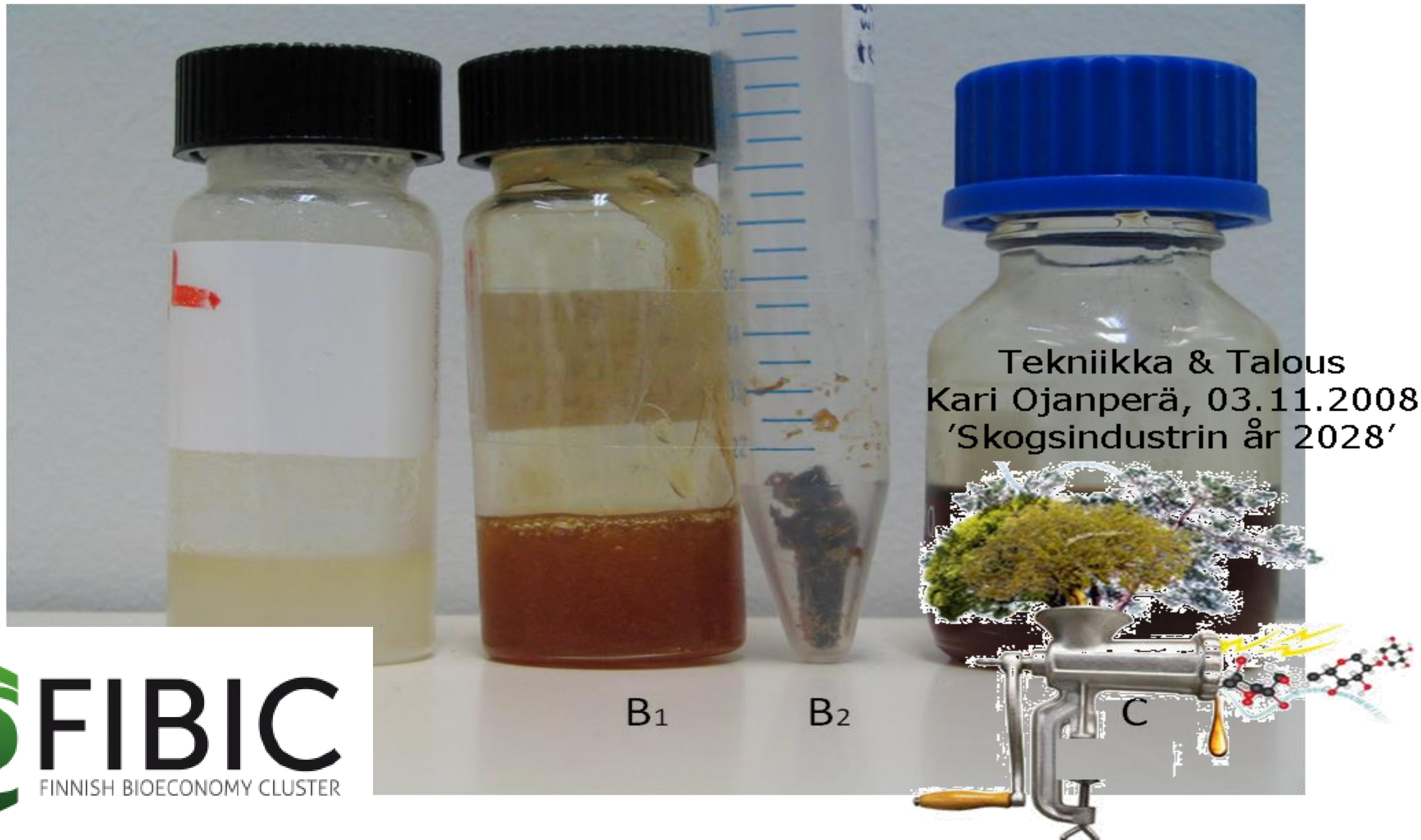
SLU

Swedish University of Agricultural Sciences

Swedish **Bio4Energy** Research & Development Platforms



But also the Finnish Bioeconomy Cluster, FIBIC was during the first years funding our research



FIBIC
FINNISH BIOECONOMY CLUSTER

..and nowadays we are also a participant in the Wallenberg Wood Science Center

..but also Umeå University & Luleå Technical University !

WWSC
WALLENBERG WOOD SCIENCE CENTER

About WWSC | Research | Networks | Publications | Newsletter

- History and background
- Mission and vision
- Governing board
- Executive committee
- Organization
- Staff
- Former members of WWSC
- WWSC Academy
- Media Activities
- Facilities
- International collaboration
- Contact

About WWSC

Wallenberg Wood Science Center (WWSC) is a joint resea[...] center at Royal Institute of Technology (KTH) and Chalmers which aims to build a material research program that can develop new material products using the Swedish forests. The base is a donation from Knut and Alice Wallenberg Foundation.

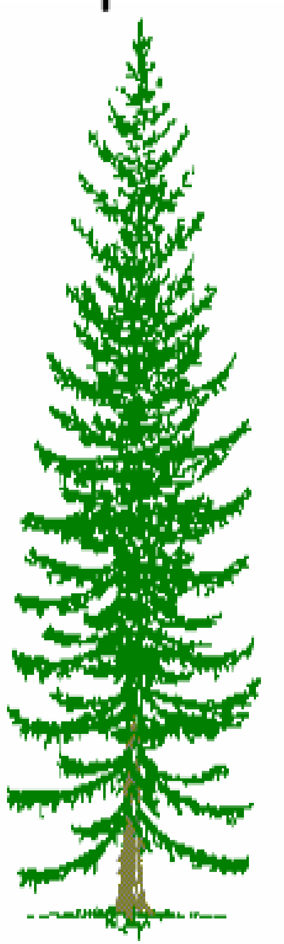
Goal: to develop a family of entirely new type of "Switchable" Ionic Liquids (SILs) and demonstrate their application in various applications

- ILs technology based on superbases: DBU, DBN, TMG (tetramethylguanidine) and other superbases…
- Easily synthesized, recovered and recycled SILs
- Biomass fractionation using recyclable switchable ionic liquids
- Biomass pretreatment with SILs
- CO_2 and SO_2 (or other acid gas) capture with SILs
- Enzyme activation in SILs

OUR BEST AND MOST IMPORTANT RESOURCE FOREST

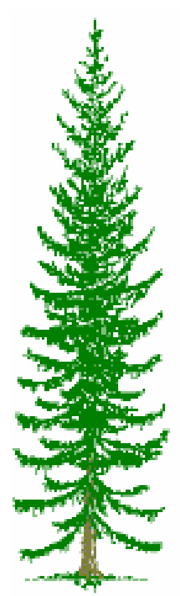
Finland

16 m³ / person year



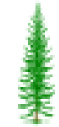
Sweden

10



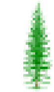
Austria

2



EU average

1.2



Why biorefining ?

- So, what do we care – oil is cheap !

Today already < 40 $ per barrel

wire.russell.com

…but do we risk that the world will be completely destabilized ?

Motivation

WHAT AFTER OIL & OTHER FOSSIL RESOURCES

Perhaps MORE E-HYPE ...but is it sustainable ?

NyTeknik en ri

Nyheter Lediga jobb Webb-tv Debatt & ledare Populär teknik 33-listan

Startsida IT & Telekom Energi Fordon Elbil Automation Innovation Bygg

Arbetare på Foxconn-fabrik i Kina. Foto: Kin Cheung / TT

En mobiltelefon ger 86 kilo avfall

rock.rapgenius.com www.greenpeace.org

But of course we can choose to solve our mobility differently in future !

permaculturenews.org

preslimo.wordpress.com

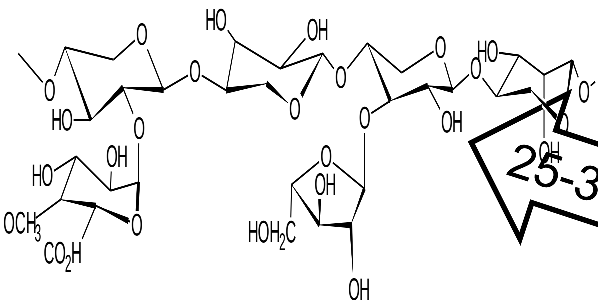
www.regionc.org

forum.xcitefun.net

OUR SUSTAINABLE PAST

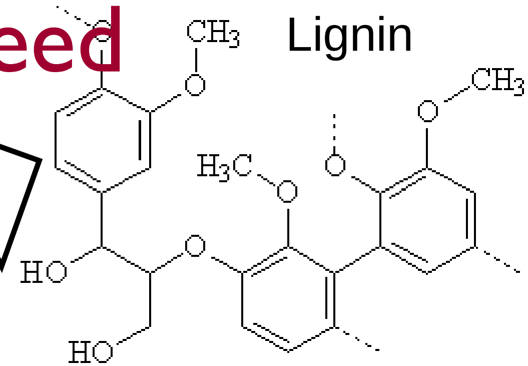
Shipping barrels of tar from ‘Tjärhovet’ at Umeå harbour around the turn of the century 1900 (photo: archives of the Nordic museum).

We need to turn back to the forest and develop tools to better transform it to all the necessities we need



Hemicelluloses

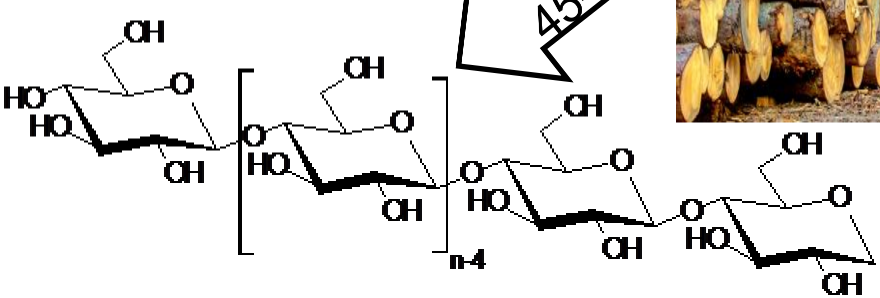
25-35%



Lignin

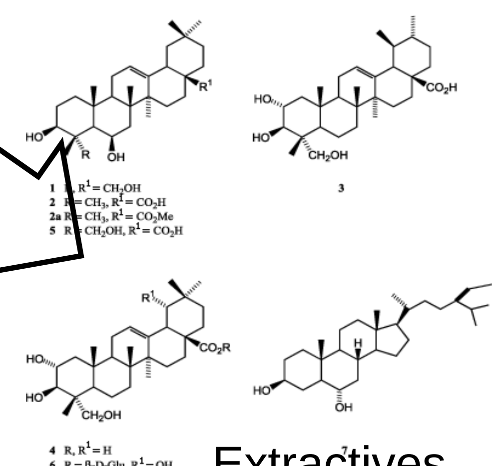
20-30%

45-50%



Cellulose

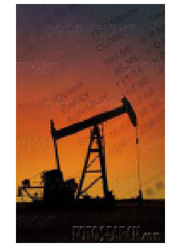
~ 5%



Extractives

BIO vs. FOSSIL RESOURCES

Biomass production 200 Gt/y
> 75 % of biomass carbohydrates



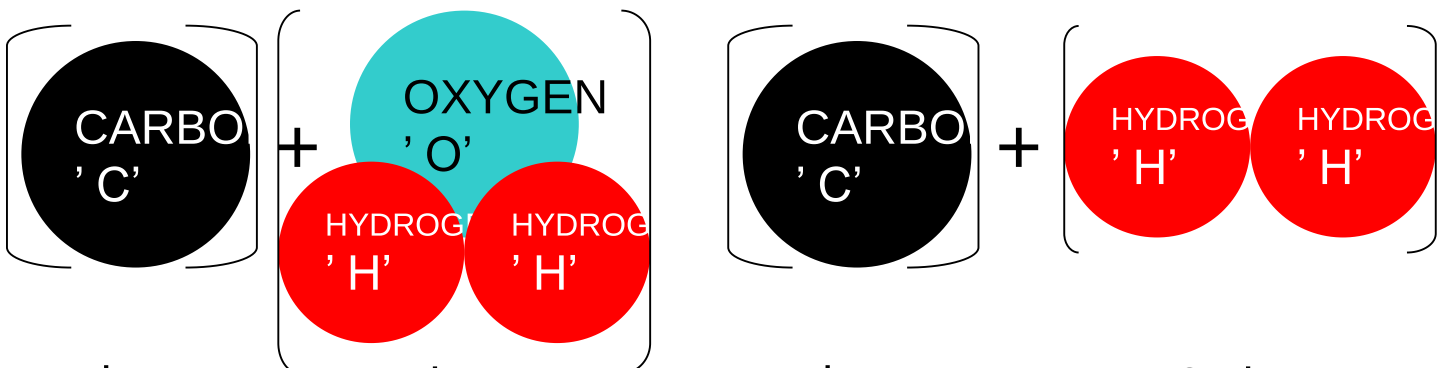
Fossil fuels extracted 7 Gt/y

Carbohydrates
$C_n(H_2O)_n$

Hydrocarbons
C_nH_{n+2}

BIOMASS

FOSSIL 'MASS'



n pieces

n pieces

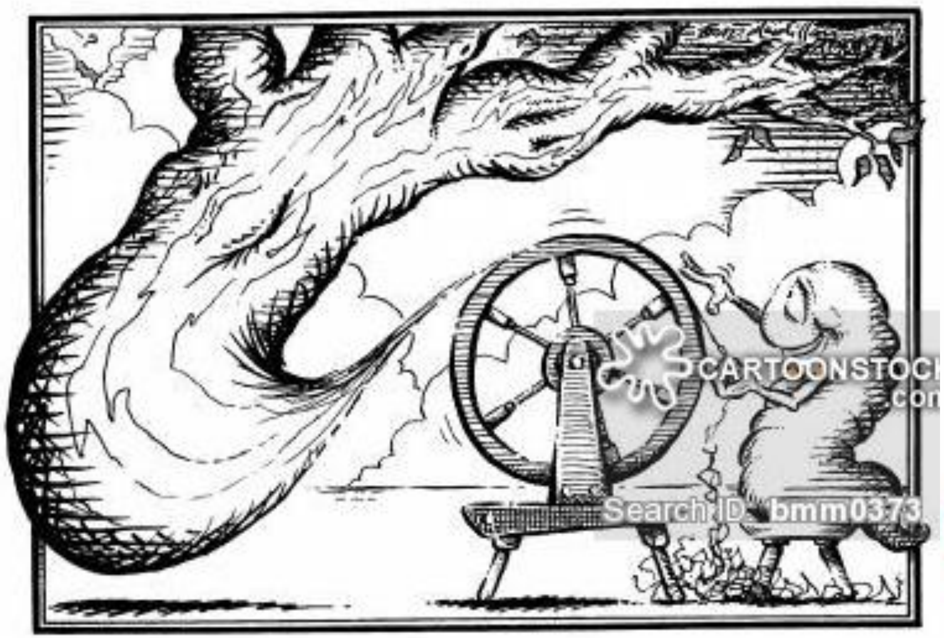
n pieces

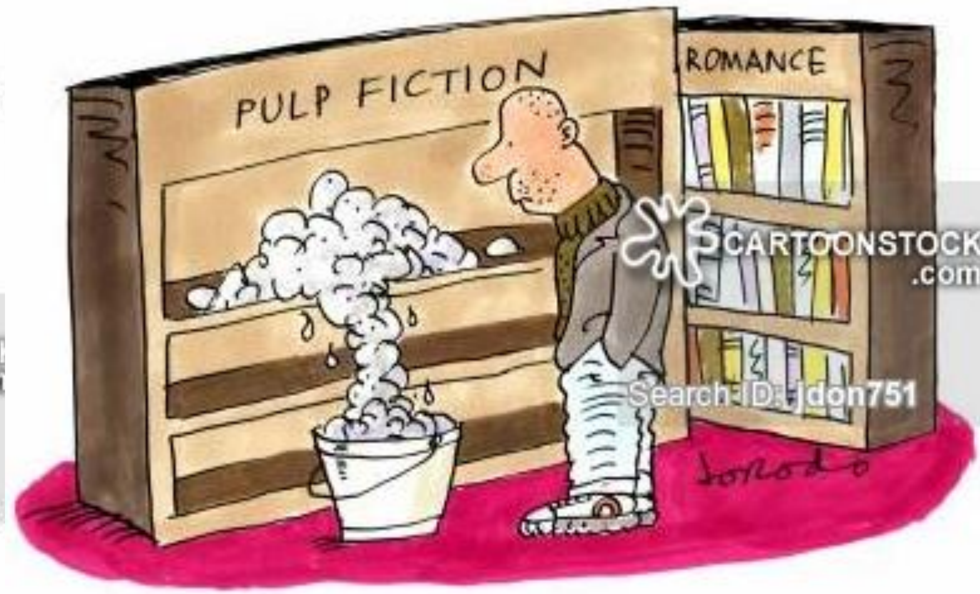
n+2 pieces

- Differences in chemical structure– technology?

So, we need new ways of converting and processing wood

- Most classical methods (fractionation) involve the use of destructive and derivative processes (e.g. kraft pulping)



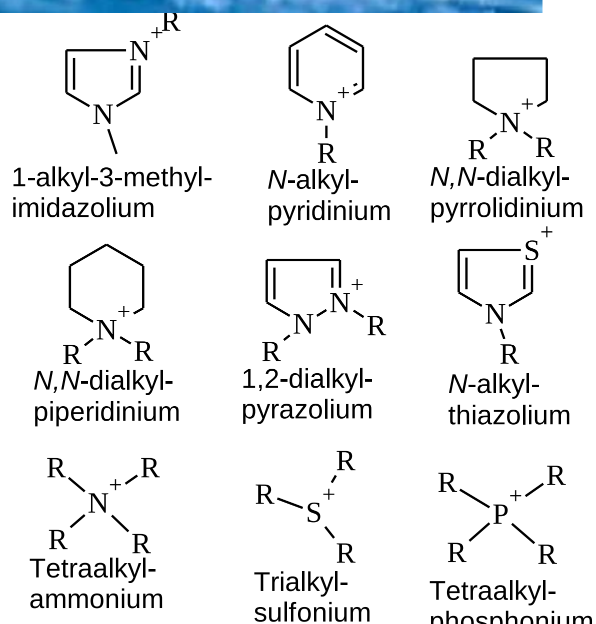


- So, let's see an alternative approach …

www3.nd.edu

Ionic liquids are :

- Ionic substances that often appear in liquid state at ambient temperatures and are composed entirely of ions.
- Frequently exhibiting almost negligible vapor pressure and flammability
- Compounds with phycochemical properties that can be tuned over an exceptional broad range just by tailoring their structure



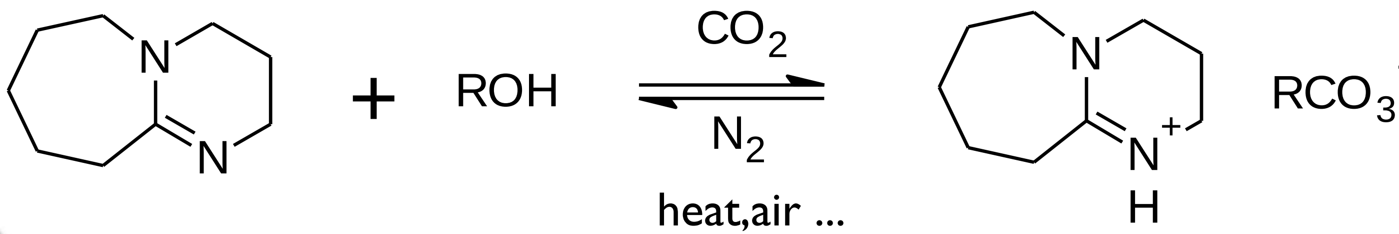
Most commonly used cations

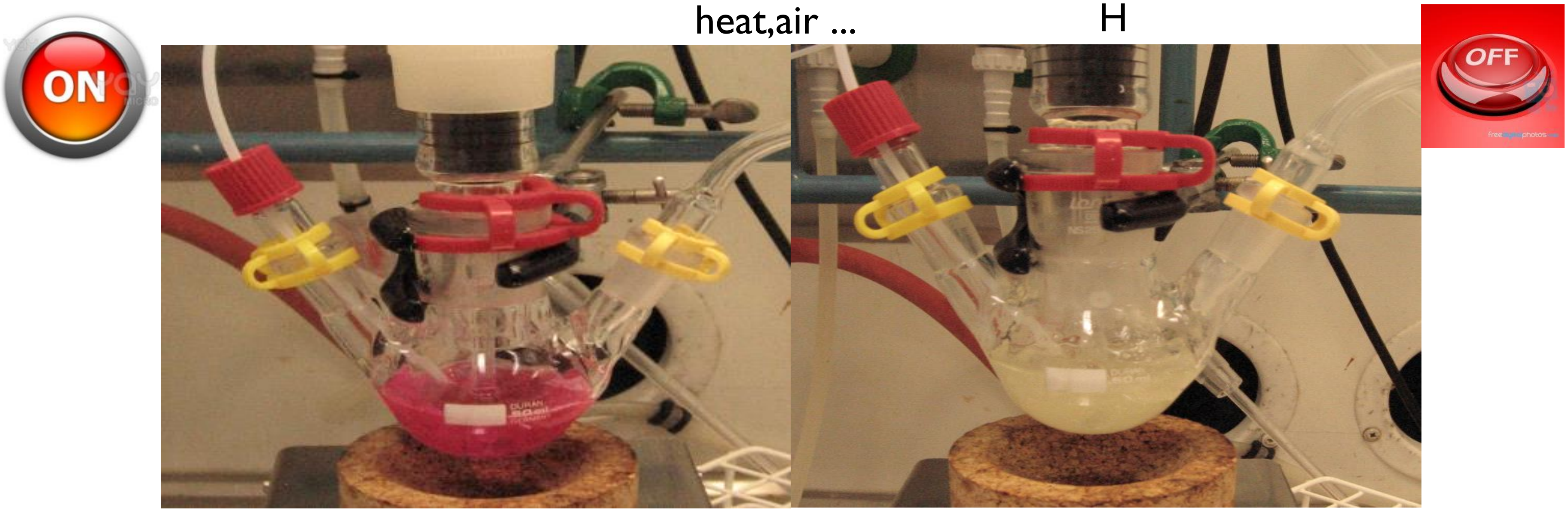
Some commonly used anions

PF_6^-	BF_4^-	$CH_3CO_2^-$
NTf_2^-	OTf^-	$CF_3CO_2^-$
$BR_1R_2R_3R_4^-$	$N(CN)_2^-$	Br^-, Cl^-, I^-

SWITCHABLE IONIC LIQUIDS

Switchable Ionic Liquids (SILs) – The IDEA





•Solvent before and after switching mixed with Nile red dye to demonstrate the transformation from low polarity to high polarity.

FIRST PROTOTYPE SWICHABLE IL:

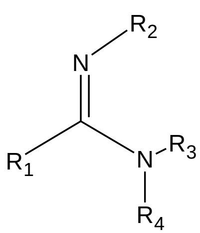
Jessop, P. G.; Heldebrant, D. J.; Xiaowang, L.; Eckert, C. A.; Liotta, C. L., Reversible non-polar-to-polar solvents. *Nature* **2005,** *436*, 1102.

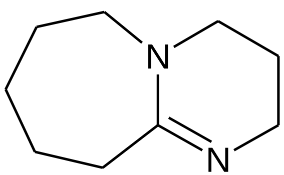
SAMPLE SIL STRUCTURES DEVELOPED BY US

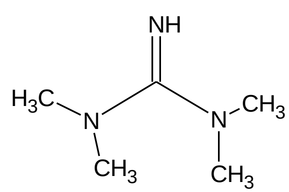
amidine, guanidine + R_1-$(R_2\text{-}OH)_x$ + acid gas → SIL

- An organic super base (DBU, TMG, BTGM...) + OH-containing compound + acid gas

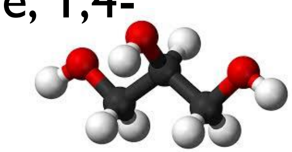
+ R_1-$(R_2\text{-}OH)_x$ + CO_2, SO_2, NO_2, CS_2,...or their mix



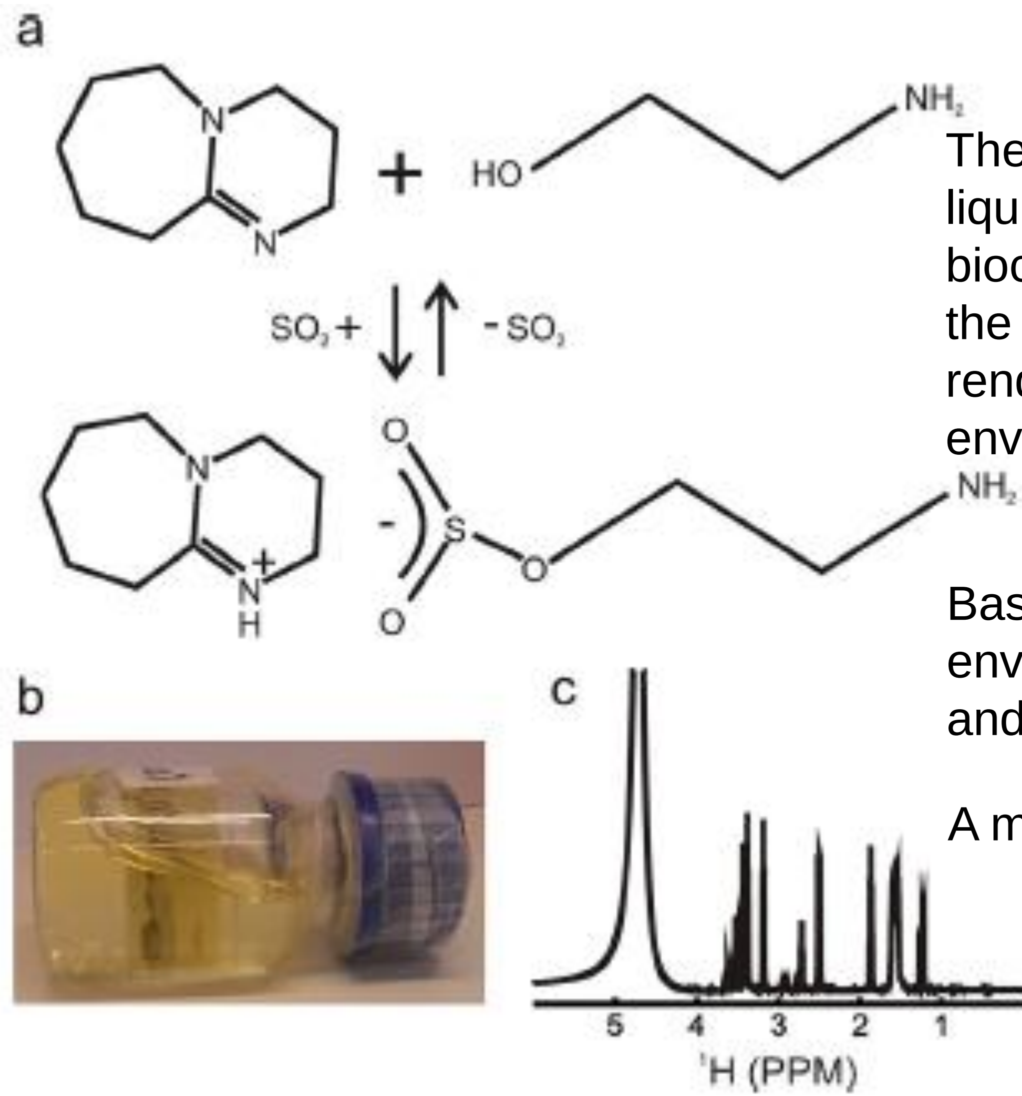




- Examples of the 'OH-compounds'
  - glycerol, choline chloride, sorbitol, xylitol, taurine, ethanol, hexanol, 2-amino ethanol, ethylene glycol, propylene glycol, diethanolamine, 1,4-butanediol (tetramethylene glycol)…



ENZYMES ENJOY THE AQUEOUS SIL ENVIRONMENT



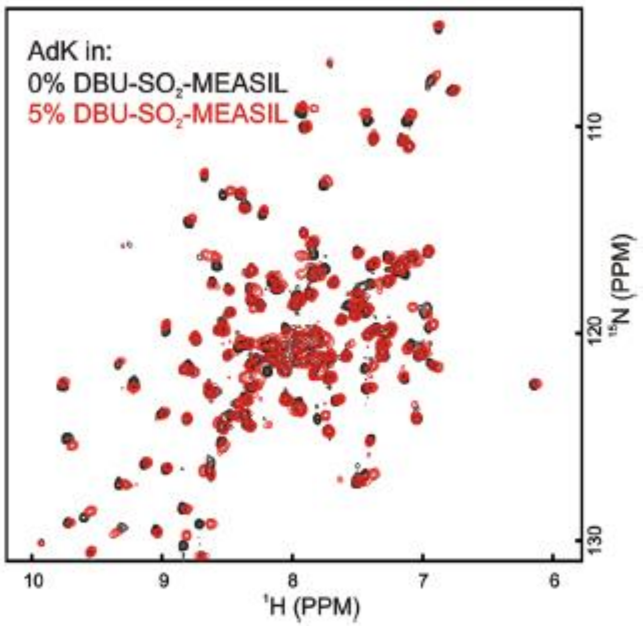
There is a growing interest in using ionic liquids to replace organic solvents in biocatalysis, both in order to improve the process efficiency and potentially render the processes more environmentally friendly

Basic knowledge of the influence of the IL environment on the structure, dynamics, and the activity of enzymes is required

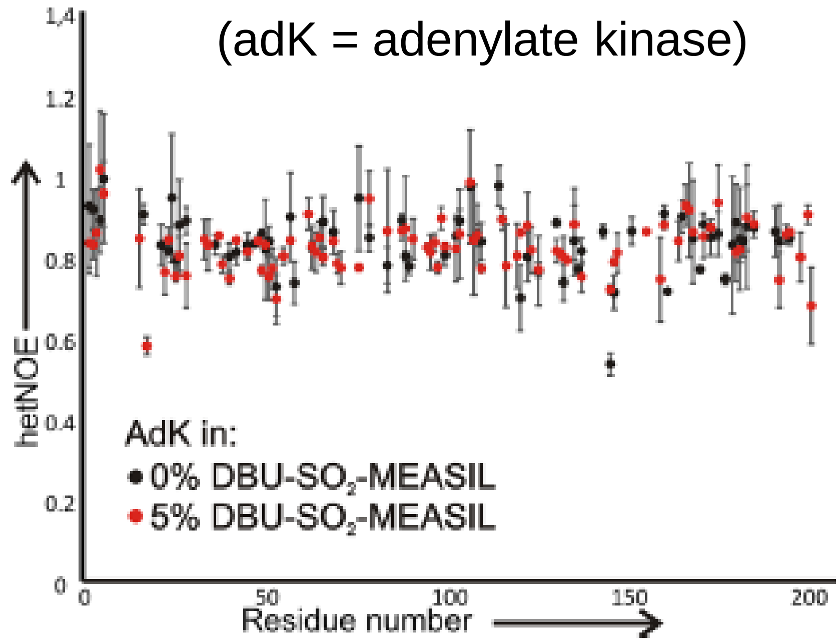
A model enzyme, adenylate kinase (AdK)

Monoethanol amine (MEA) and 1,8-diazabicyclo-[5.4.0]-undec-7-ene (DBU) derived SIL with sulfur dioxide (SO_2) as the coupling media (DBU-SO2-MEASIL).

ENZYMES ENJOY THE AQUEOUS SIL ENVIRONMENT



^1H-^{15}N-HSQC spectrum of thermostable adenylate kinase from *Aquifex aeolicus*



Taken into account both the chemical shift perturbation and the spin-relaxation data, it is evident that both the structure and ps–ns dynamics of AdK are very similar in water and in SIL solutions.

The spectra are well-dispersed at all concentrations of the switchable ionic liquid

A lot of NMR time was spent on our state-of-the-art 850 Mhz machine

The signals from the three different nucleotides are well separated in ^{31}P NMR spectra and the concentration of each individual species can be monitored directly during catalysis

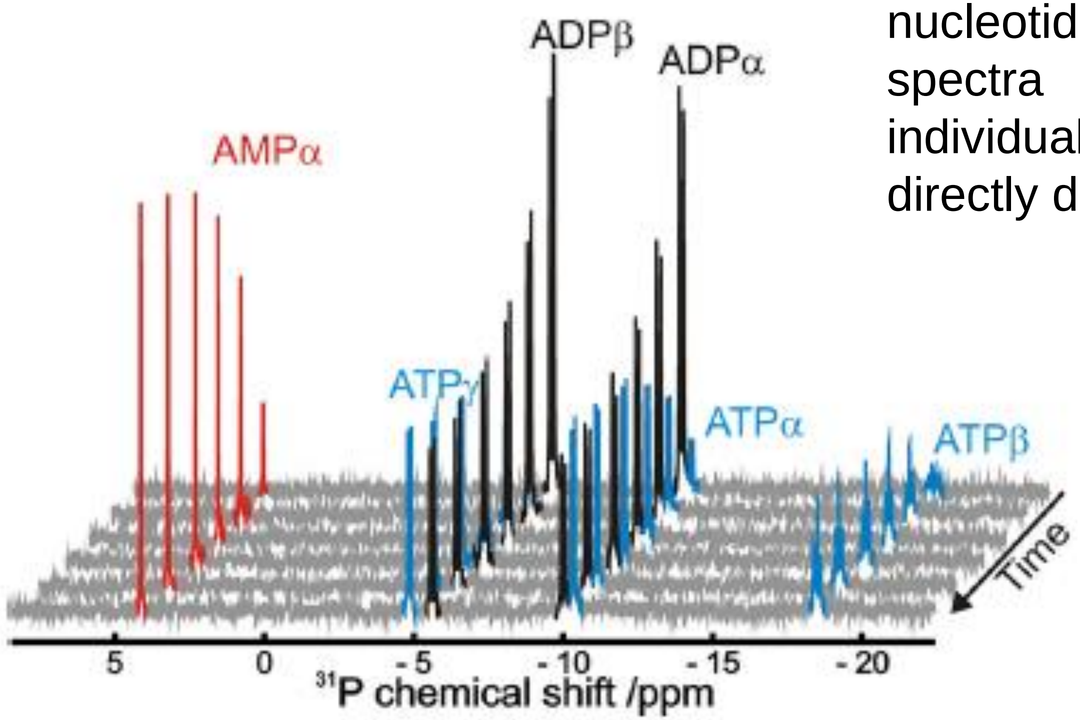


Figure 5. NMR spectra of AMP (red), ADP (black), and ATP (blue) at time intervals of 27 min from the addition of thermostable adenylate kinase (*Aquifex aeolicus*) to a sample containing 17 mM ADP and 15 wt % ionic liquid at 50°C. The assignments was obtained from Cohn et al.[9]

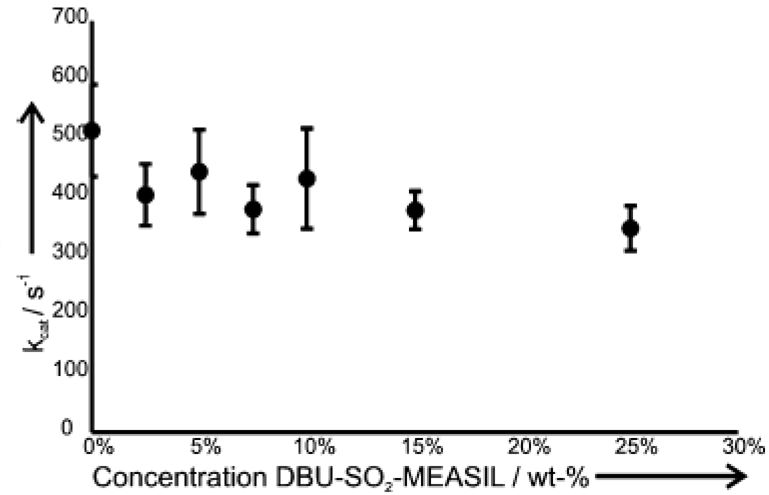


Figure 7. The measured catalytic turnover number for the adenylate kinase as a function of the concentration of DBU-SO_2-MEASIL. The rates were measured at 50 °C, at an initial concentration of 17 mM of ADP. For each concentration of DBU-SO_2-MEASIL three replicates were measured. The error bars indicate the standard deviation of the three replicates.

The results indicate that DBU-SO2-MEASIL is not interfering with or binding to the active site

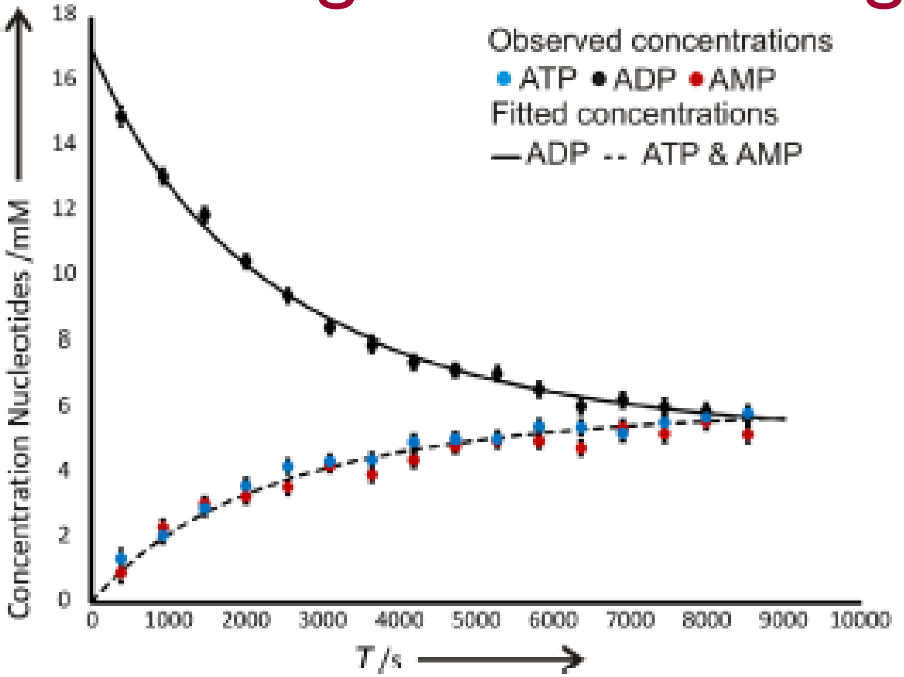


Figure 6. Concentrations of the three nucleotides AMP (red), ADP (black), and ATP (blue), measured with ^{31}P NMR spectroscopy, as a function of time

... and hard and softwood pulp can also be easily produced with the SIL technology – in 2 hours only!

Summary

- By using the new ^{31}P NMR –based real-time assay, we show that DBU-SO_2-MEASIL is compatible with enzymatic catalysis
- The (aqueous) SIL systems can also solubilize biomass, including lignocellulose (e.g. wood) thus providing avenues for new processes (e.g. enzymatic degradation of hemicellulose)
- SIL is a promising process solvent e.g. in cases when treatments with ionic liquids are followed by an enzymatic process.
- We are currently developing yet new SILs and will also study their compability with enzymes and yeasts

Biofuels production

First generation

Ethanol from Fermentation:

$$\text{Sugar(s)} \xrightarrow{\text{Microorganisms}} \text{Ethanol + By-products}$$

$$\underset{\text{(starch)}}{(C_6H_{10}O_5)_n} + nH_2O \xrightarrow{\text{enzymes}} \underset{\text{(glucose)}}{nC_6H_{12}O_6}$$

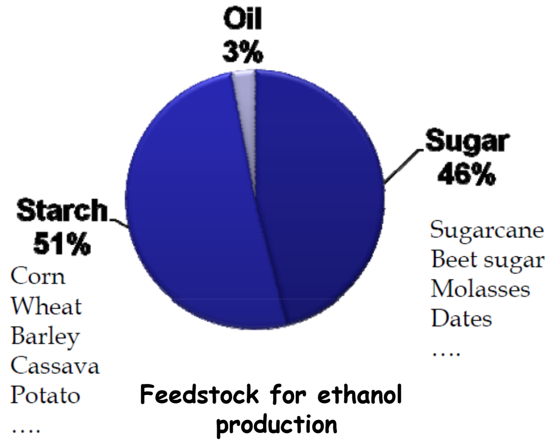
$$\underset{\text{(glucose)}}{C_6H_{12}O_6} \xrightarrow{\text{yeast}} \underset{\text{(ethanol)}}{2CH_3CH_2OH} + 2CO_2 + \text{heat}$$

$$\underset{\text{Sugars (e.g. glucose)}}{C6H12O6} \xrightarrow{\text{Microorganisms}} \underset{\text{(Butanol)}}{C4H9OH} + 2CO2 + H_2O$$

Synthetic ethanol:

Oil ⟶ Ethylene ⟶ Ethanol

$$C2H4 + H2O \Longleftrightarrow C2H5OH$$



Feedstock for ethanol production

Source: www.asiantrendsmonitoring.com

"...large increases in biofuels production is the main reason behind the steep rise in global food prices" -World Bank policy research working paper July 2008"

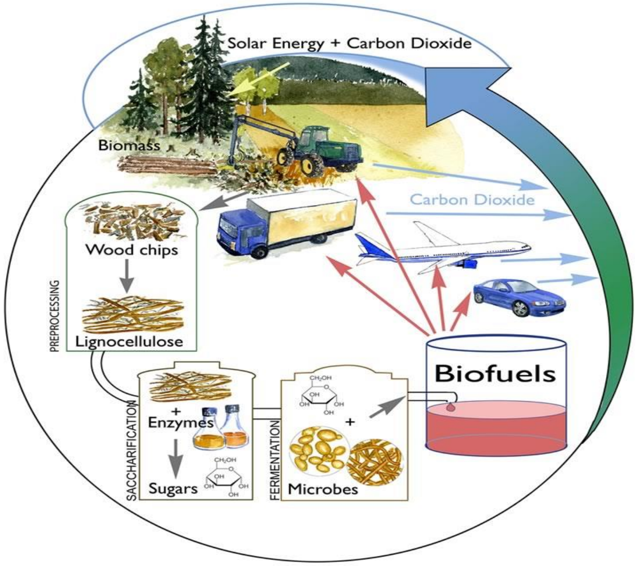
Lignocellulosic materials

Forest residues

Agricultural residues

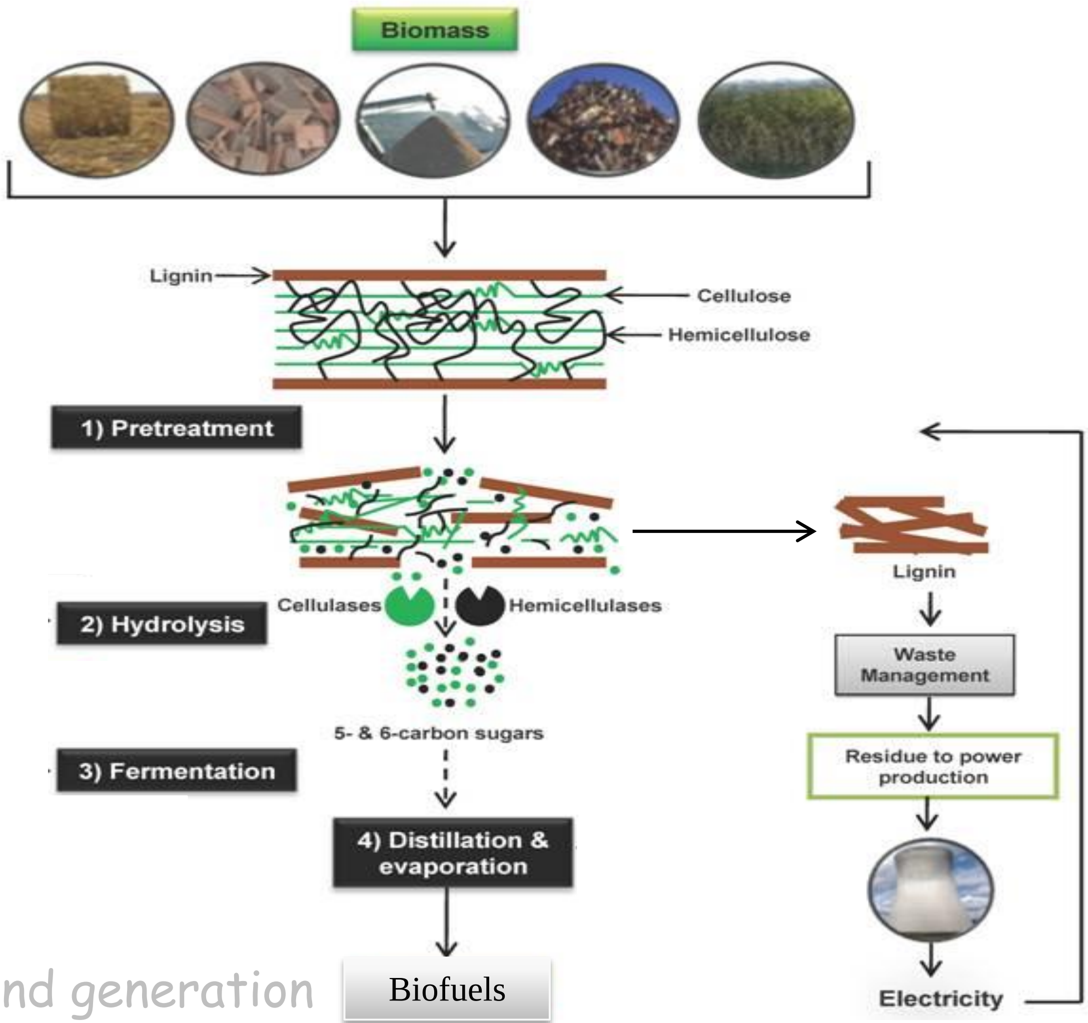
Municipal solid wastes

- Abundant
- Inexpensive
- Sustainable and potential feed stocks



Source: www.bioimprove.se

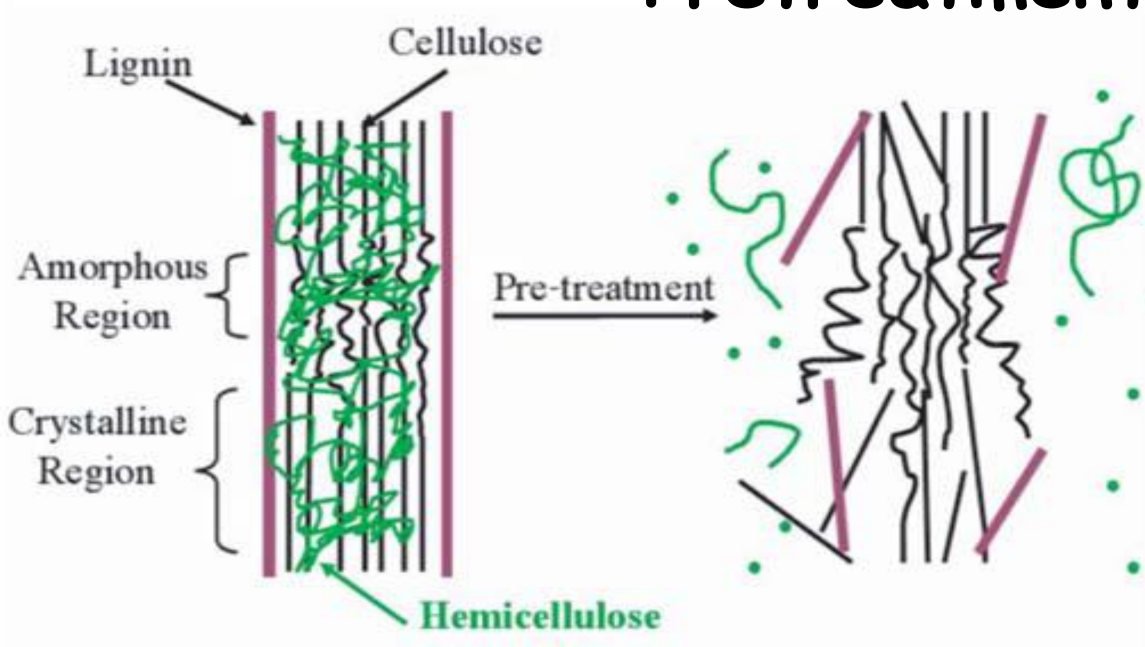
Cellulosic fules production



Second generation

"Cellulosic fuels can achieve much greater energy efficiency and GHG benefits"

Pretreatment



Goals:

- Opening crystalline structure of Lignocellulose
- Hydrolysis of hemicelluloses
- Enhance the enzymatic hydrolysis

IL's: Novel alternative green solvents for the pretreatment of lignocelluloses.

Physical methods	Physical-chemical Methods	Chemical Methods	Biological methods
➢ Mechanical Pre-treatment. ➢ Pyrolysis. ➢ Microwave irradiation	➢ Steam explosion. ➢ Liquid hot water (LHW). ➢ Ammonia fiber explosion (AFEX). ➢ CO2 explosion.	➢ Ozonolysis. ➢ Dilute-acid hydrolysis. ➢ Concentrated-acid hydrolysis. ➢ Alkaline hydrolysis. ➢ Oxidative delignification. ➢ Wet oxidation. ➢ Organosolv process. ➢ Dissolution in IL's	➢ Fungal pretreatment.

Hydrolysis

Splitting of cellulose to glucose by

– Concentrated acid hydrolysis at room Temp. by e.g.

  » 72% H_2SO_4

  » 42% HCl

– Dilute acid hydrolysis

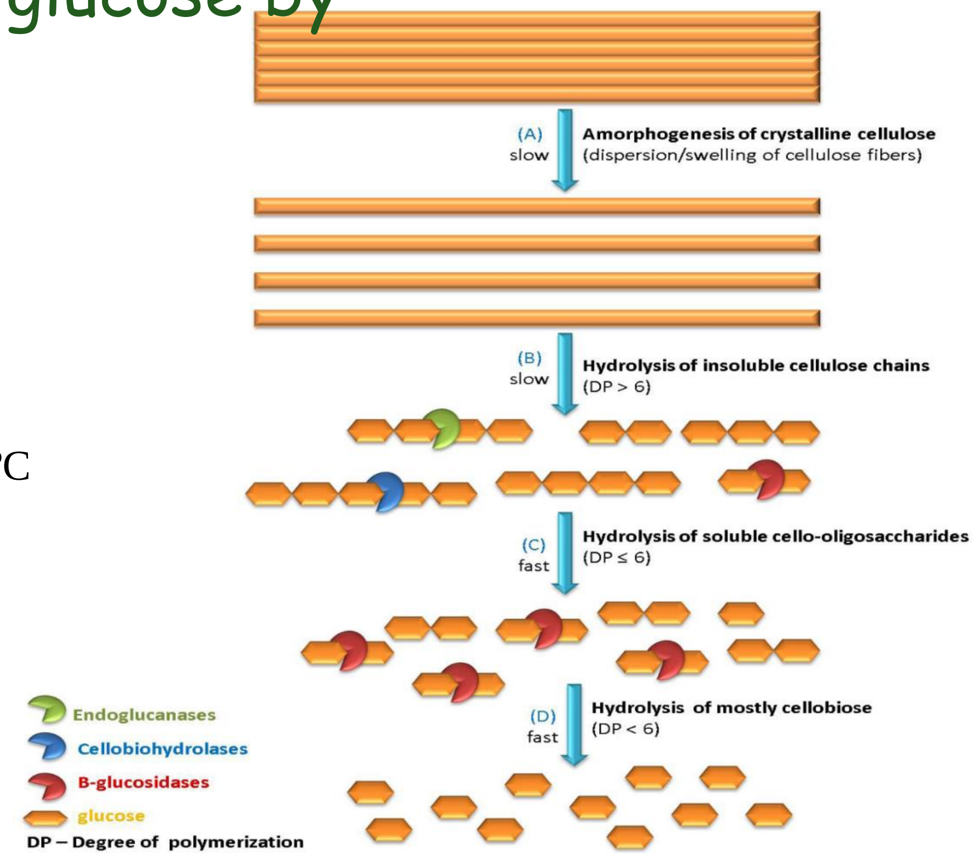
  » e.g. 0.5% sulfuric acid at 200-220ºC (ca 20-25 bar), 5-10 min

– Cellulases enzymes at ca 45°C and 24-72 hours

  » endoglucanases,

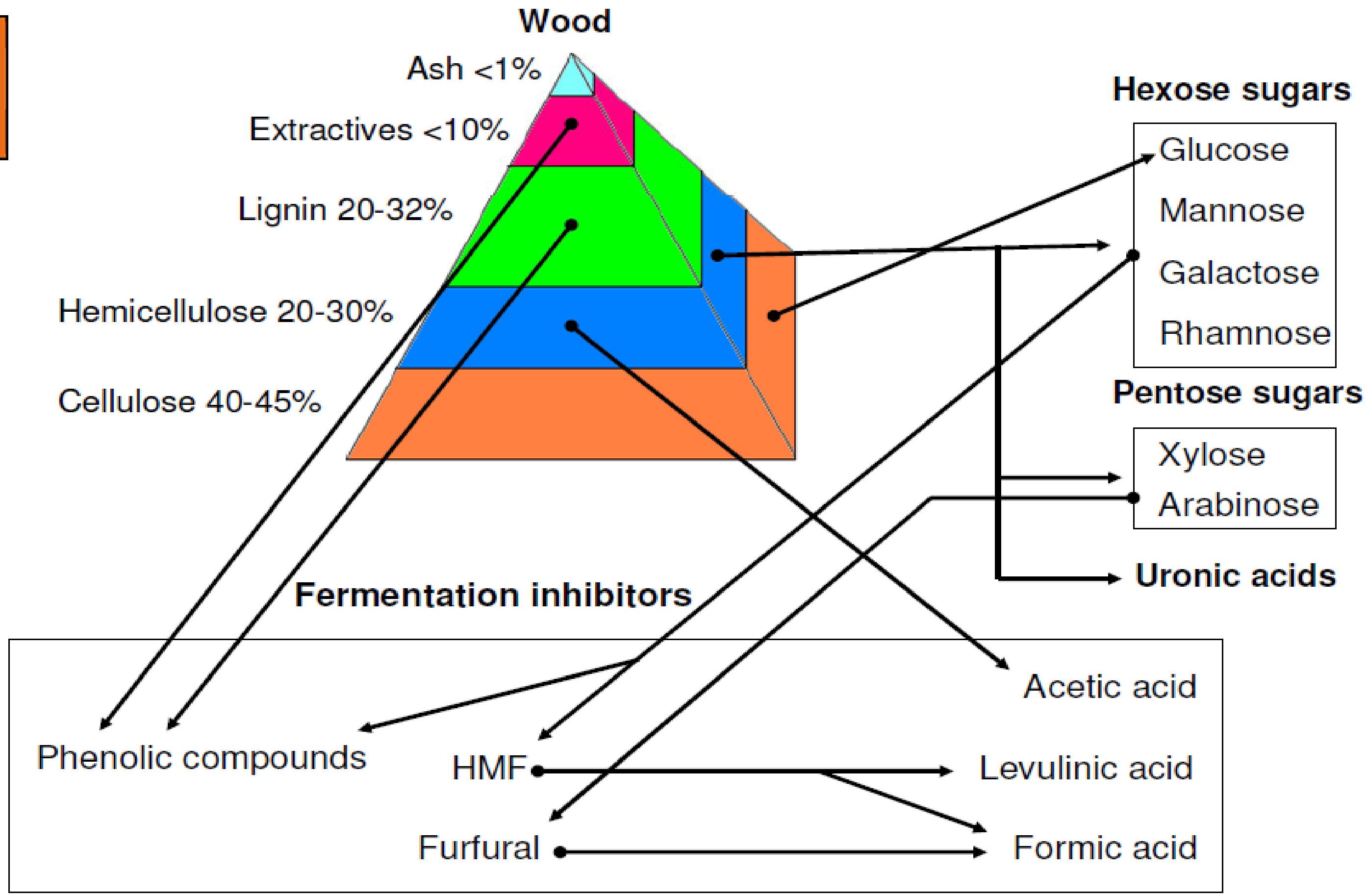
  » exoglucanases,

  » ß-glucosidases



Source: Arantes et al., 2010

Toxic compounds from Pretreatment



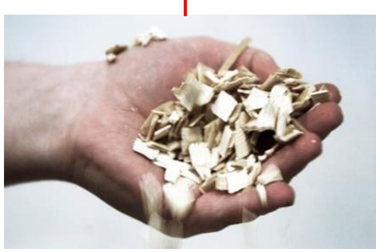
(Source: Alriksson B., 2006)

Enzymatic hydrolysis of softwood and agricultural residues after treatment with ionic liquid (1-allyl-3-methylimidazolium formate)

Thermochemical pretreatment of sugarcane bagasse and Norwegian spruce was performed in the Örnsköldsvik demo plant in Sweden (<www.sekab.com>).

Sugarcane bagasse

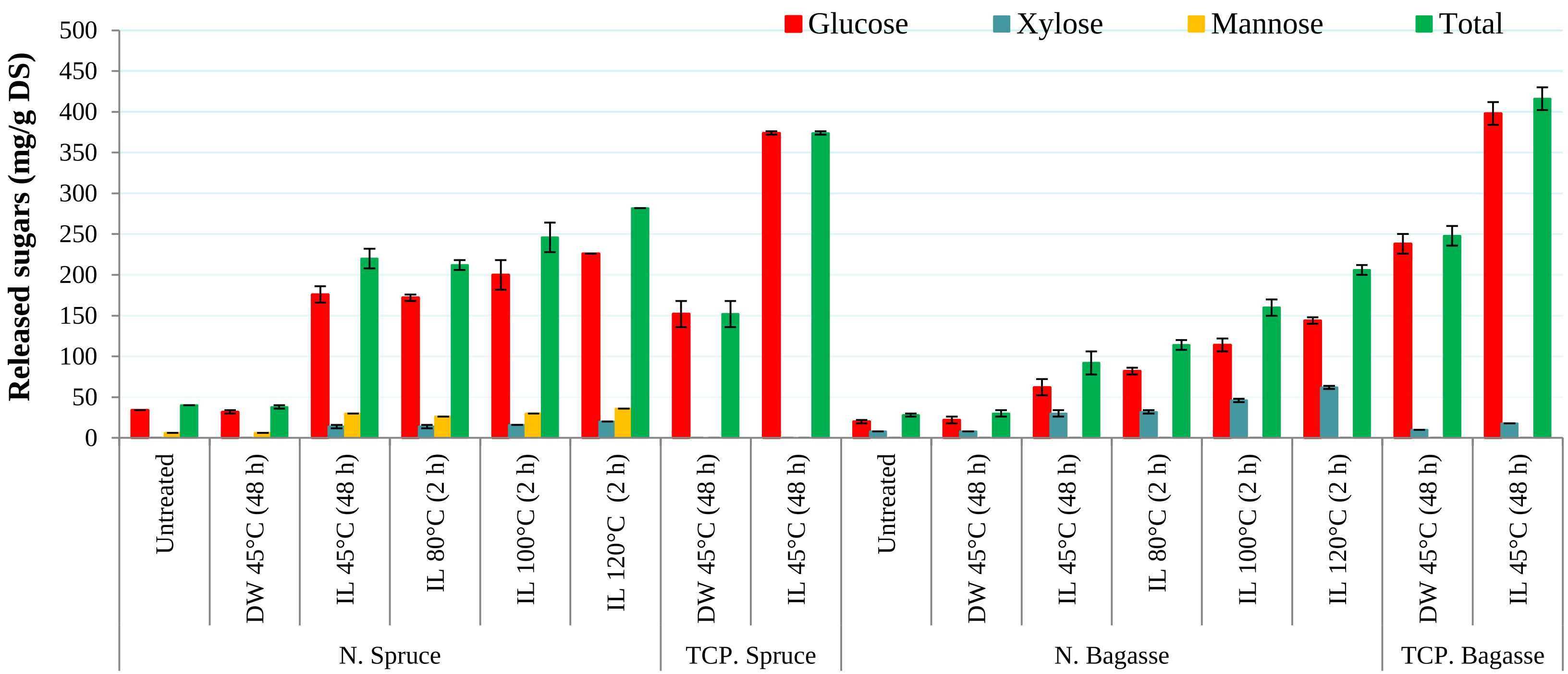
Microcrystalline cellulose



Spruce wood

Solvation of cellulosic material in ionic liquid. The figure shows 5% (w/w) cellulose dissolved in [AMIM][Fo].

Enzymatic hydrolysis of Norway spruce and sugarcane bagasse after treatment with Ionic Liquid



Material, temperature and time of IL and DW pretreatment

Monomer sugars obtained after enzymatic hydrolysis (72 h at 45 ºC) of native (N) and thermo – chemically pretreated (TCP) spruce and sugarcane bagasse. The cellulosic substrates were treatment with [AMIM]Fo ionic liquid (IL) or deionized water (DW).

Enzymatic hydrolysis of softwood and agricultural residues after treatment with ionic liquid

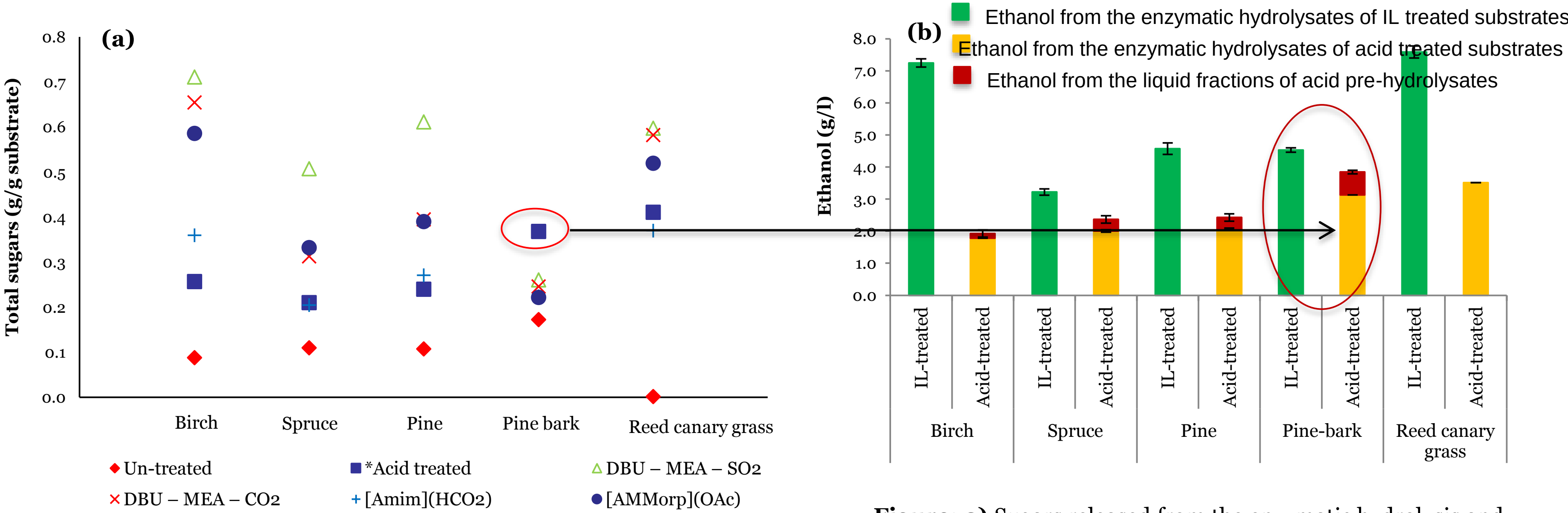
Venkata Prabhakar Soudham, John Gräsvik, Björn Alriksson, Jyri-Pekka Mikkola, Leif J. Jönsson

Lignocellulose

- A new stratagy was developed to lighten the toxicity of acid pre-hydrolysed lignocellulose substrates. (*Soudham et al., 2014*)


Bioresource Technology 166 (2014) 559–565
Contents lists available at ScienceDirect
Bioresource Technology
journal homepage: www.elsevier.com/locate/biortech
Detoxification of acid pretreated spruce hydrolysates with ferrous sulfate and hydrogen peroxide improves enzymatic hydrolysis and fermentation
Venkata Prabhakar Soudham [a,d], Tomas Brandberg [b], Jyri-Pekka Mikkola [a,c], Christer Larsson [d,*]

- Ionic Liquid's (IL's) dissolution of different lignocelluloses

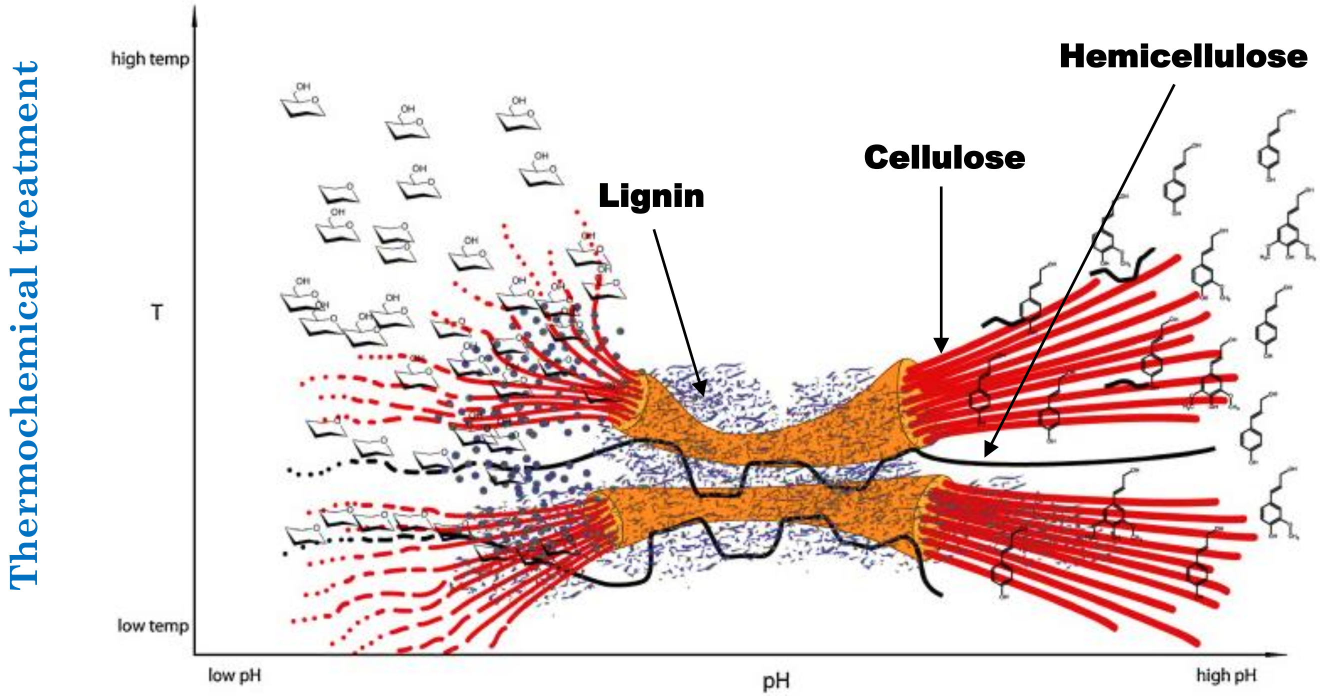


*Acid treated: total sugars released from the both acid and enzymatic hydrolysis of the substrates.

Figure: a) Sugars released from the enzymatic hydrolysis and **b)** ethanol produced from the hydrolysates of different lignocellulose substrates treated with various solvents.

Biomass pretreatment

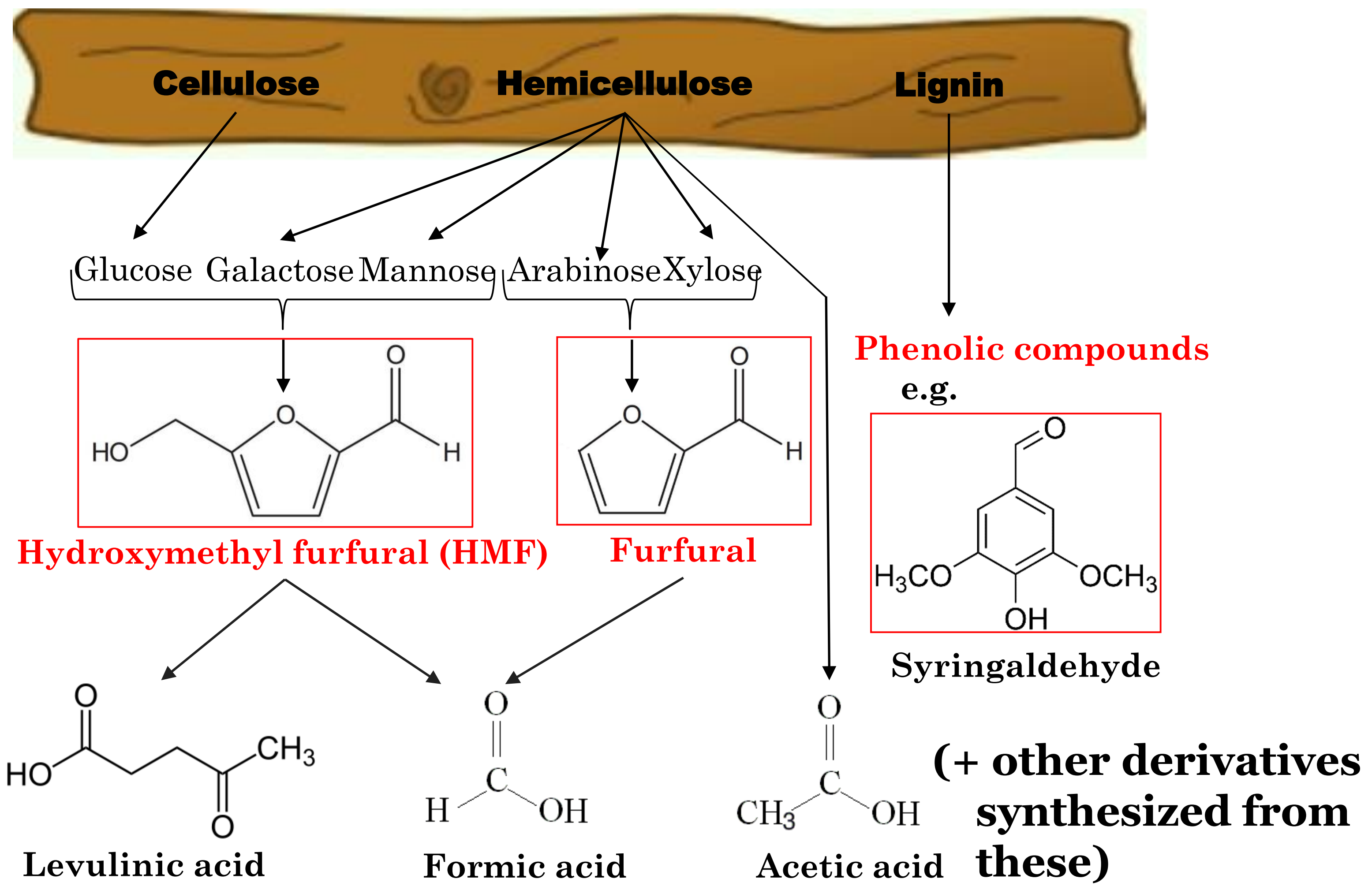
Removing the recalcitrant barriers of biomass



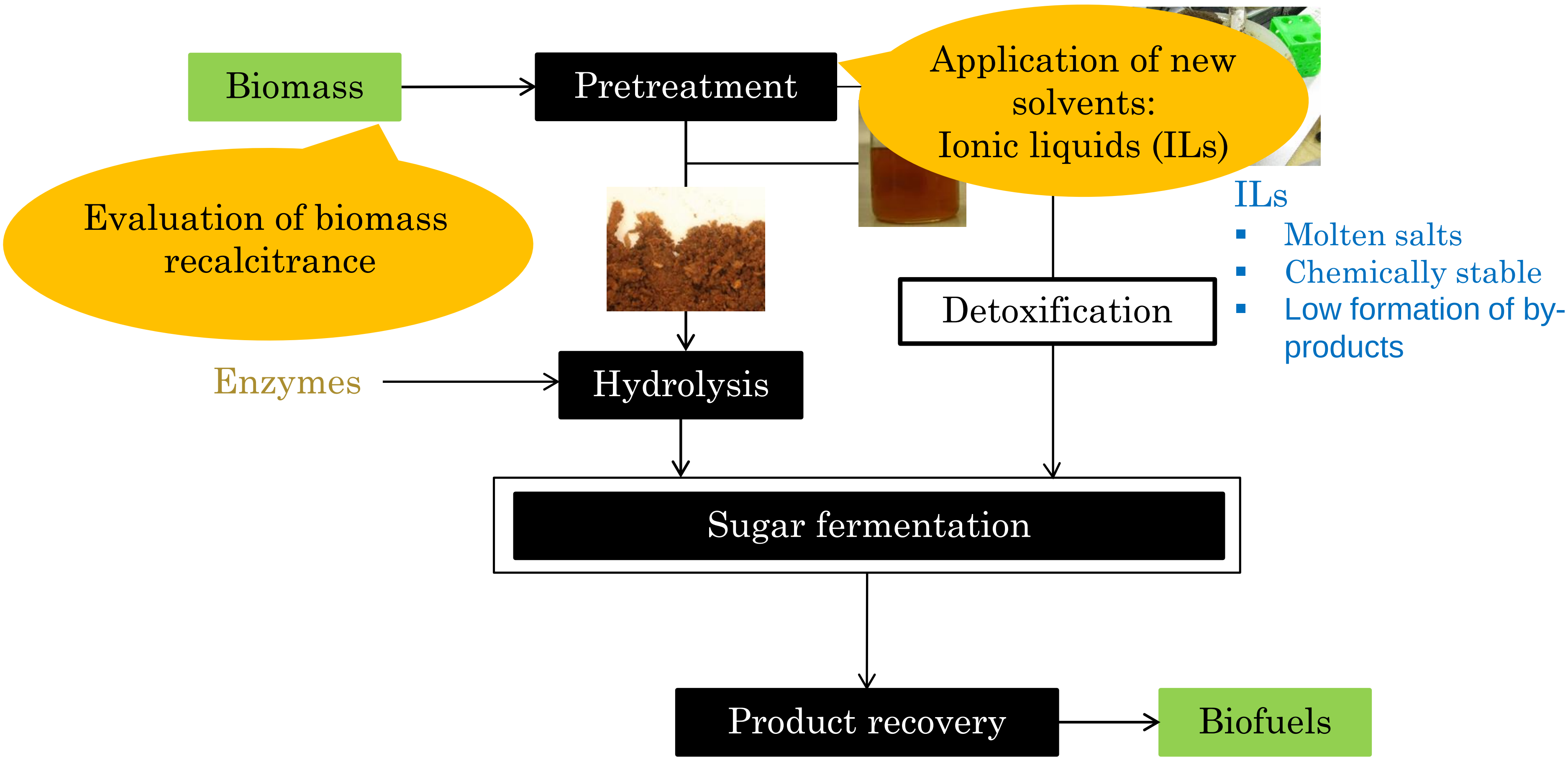
source: Pedersen and Meyer 2010

$$\text{Severity factor (SF)} = \log\left(t.\exp\left(\left(T - T_{ref}\right)/14.75\right)\right)$$

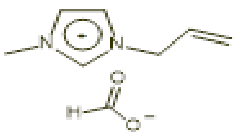
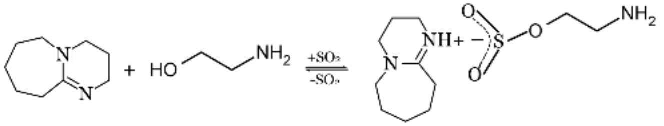
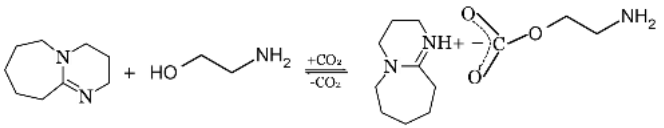
Degradation products of pretreatment–inhibitors



Critical Areas of Research



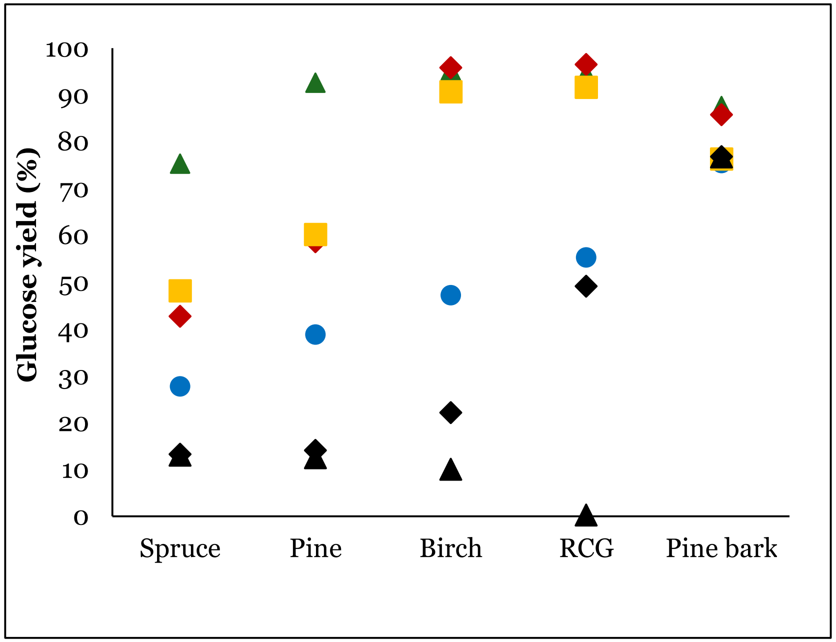
Ionic liquid (IL) solvents for the pretreatment of biomass

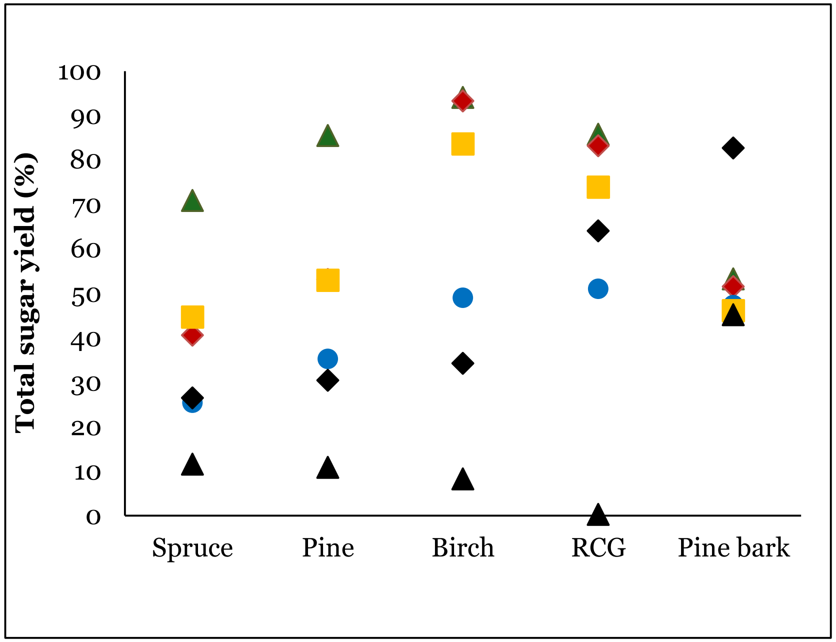
IL Name	Abbreviation	Structure
1-allyl-3-methylimidazolium **formate**	[Amim][HCO_2]	
N-allyl-N-methylmorpholinium **acetate**	[AMMorp][OAc]	
DBUH+ [MEA- Sulfonate]$^-$	**SO_2** DBU- MEA SIL	
DBUH+ [MEA- Carbonate]$^-$	**CO_2** DBU–MEA SIL	

DBU - 1,8-Diazabicycloundec-7-ene

MEA - Monoethanolamine

IL treatment of native lignocelluloses





Sugars released from the 48 h enzymatic hydrolysis of (▲) non-treated, (♦) acid treated, and IL treated (▲) **SO2 DBU- MEA SIL**, (♦) **CO2 DBU–MEA SIL**, (■) **[AMMorp][OAc]**, and (•) **[Amim][HCO2]** lignocelluloses materials.

Åbo Akademi University

Superior Process for Cellulose Nanofibres from Switchable Ionic Liquid Treated Wood Chips

Ikenna Anugwom, Linn Berglund, Yvonne Aitomäki, Kristiina Oksman and Jyri-Pekka Mikkola

Typical STHT parameters

- Wood chips are treated with DBU-MEA-SO_2 SIL for **2 h** at 160°C
- Wood-to-SIL wt-ratio 1:5
- No stirring
- Water addition to the SIL (37 wt-%)
- In a pressure vessel - no decomposition of the SIL due to the gas equilibrium in the system

Short time high temperature (STHT)

- Short treament time
- Still milder conditions than e.g. Kraft pulping
- Ease of handling
- Pre-treatment could be avoided
    - Avoid excessive drying
    - Avoid excessive milling (no degradation)
    - Avoid multiple pretreatment steps

Hard and Softwood- Results

Birch treated by the STHT approach

Composition	Amount (%)
Cellulose	72
xylan	19
Other sugars	2.1
Lignin	4.8

Spruce treated by the STHT approach

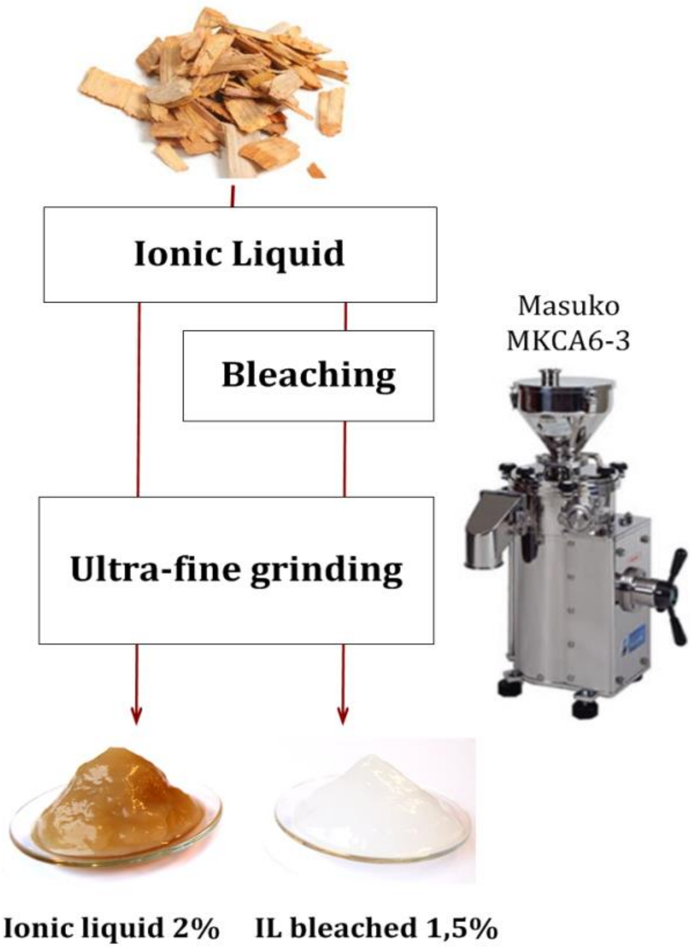
Composition	Amount (%)
Cellulose	75
xylan	2.7
Gluco man	7.2
Other sugars	1.5
Lignin	5.8

Cellulose Nanofiber: AIM

- To evaluate the separation of SIL treated wood chips from birch by ultra-fine grinding. The reference material used was birch Kraft pulp.
- Using viscosity measurements and optical microscopy to assess the fibrillations during the grinding process.
- To evaluate the degree of separation and mechanical properties of dried fiber networks.
- Characterizing size distribution of the nanofibers using atomic force microscope (AFM)
- To evaluate energy consumption required for the grinding process

Materials:

- Bleached Kraft pulp from birch (reference material)
- SIL treated birch wood chip before bleaching
- SIL treated birch wood chip bleached (Chlorite bleaching)



Characterization

Chemical analysis: Gas chromatography

Viscosity: A sine-wave Vibro Viscometer SV-10 series

Microstructure: Nikon Polarizing microscope, Eclipse LV100N POL

Mechanical testing: AG-X tensile testing machine, Shimadzu

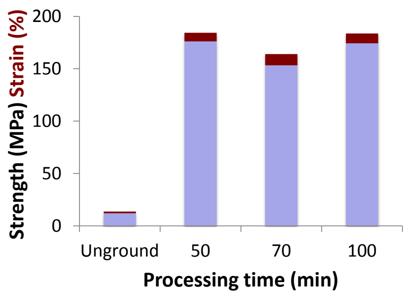
Energy consumption: Total electric power intake was measured with a power meter

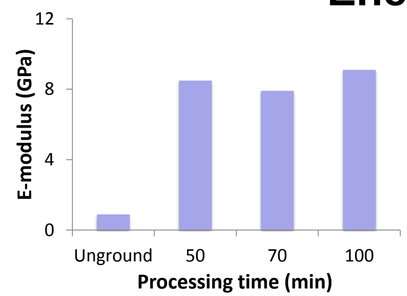
Nanostructure: AFM nanoscope V Microscope, Veeco Instrument Inc, in tapping mode

Mechanical properties of the dried fibre network

The figures shows the mechanical properties against the processing time and the measured energy demand for the mechanical treatment

Energy consumption

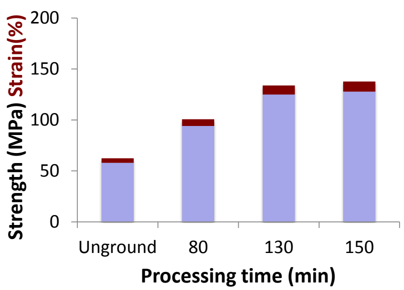


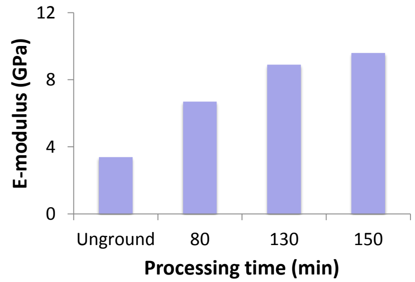


Bleached Kraft

Grinding: 100 min

13 MWh/t

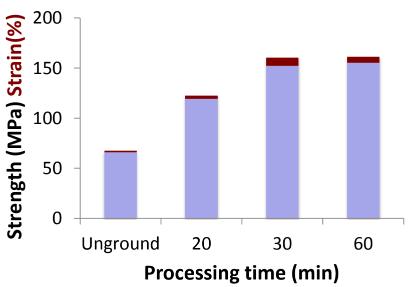


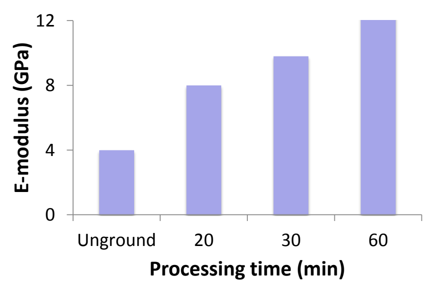


Ionic liquid

Grinding: 150 min

9 MWh/t





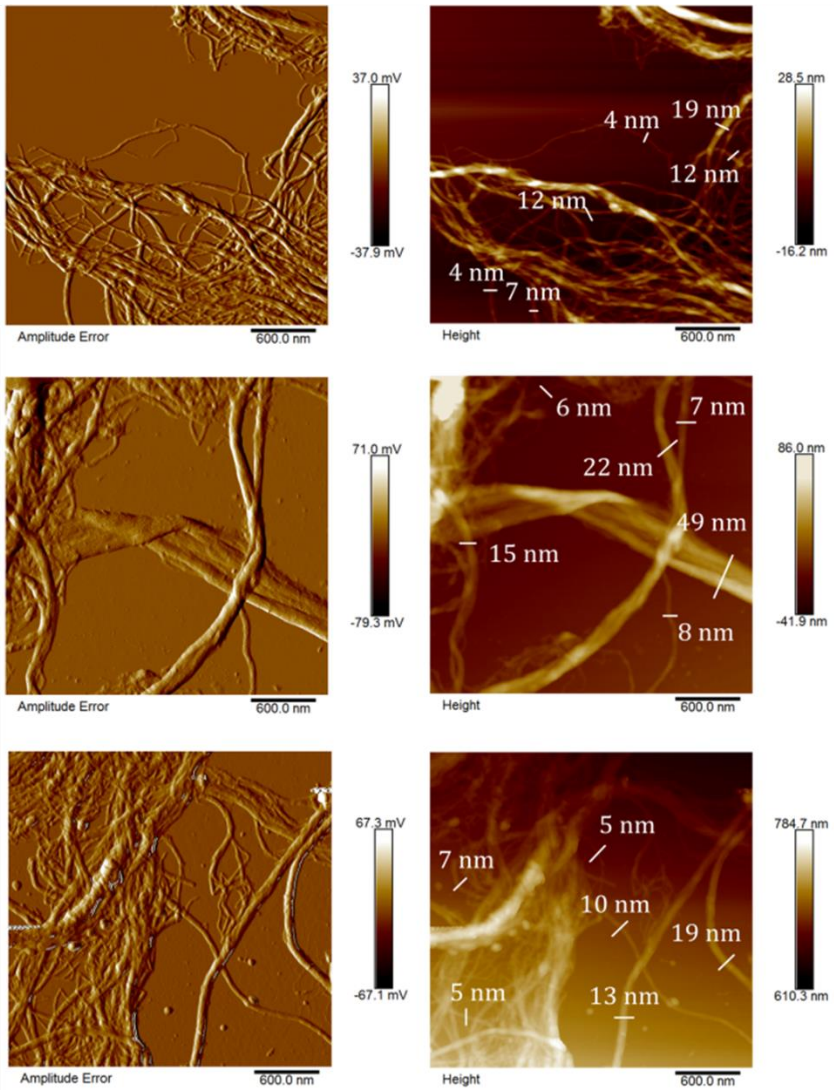
IL Bleached

Grinding: 60 min

7 MWh/t

Microscopy

The AFM amplitude and height images below shows the size distribution of the nanofibres after mechanical separation



Bleached Kraft

4-31 nm

Ionic liquid

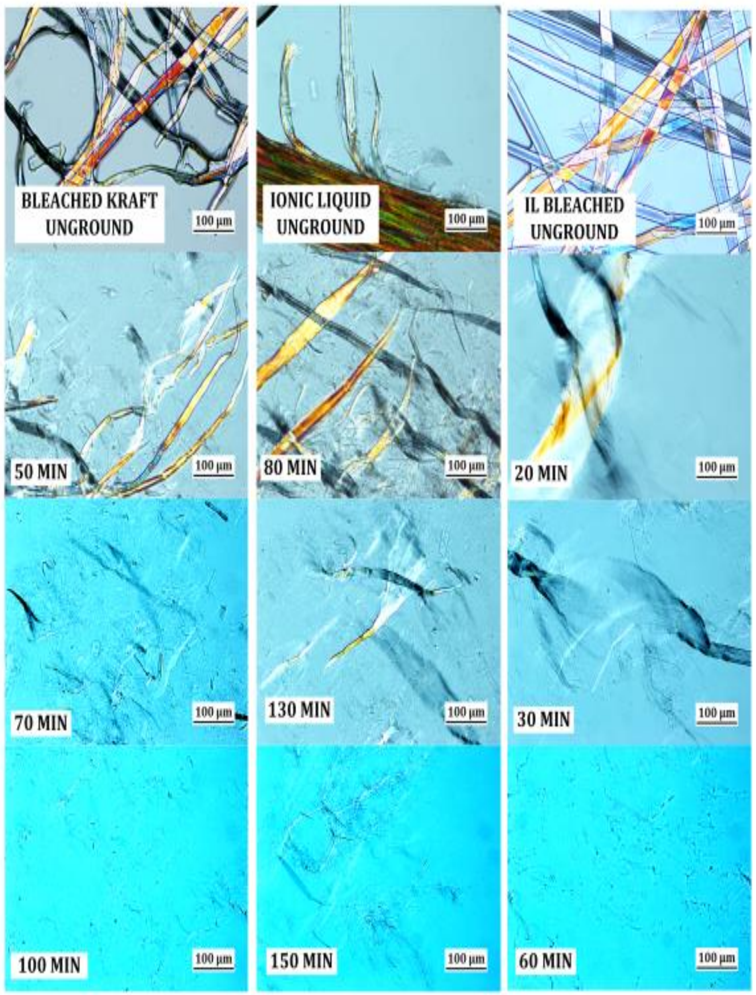
6-49 nm

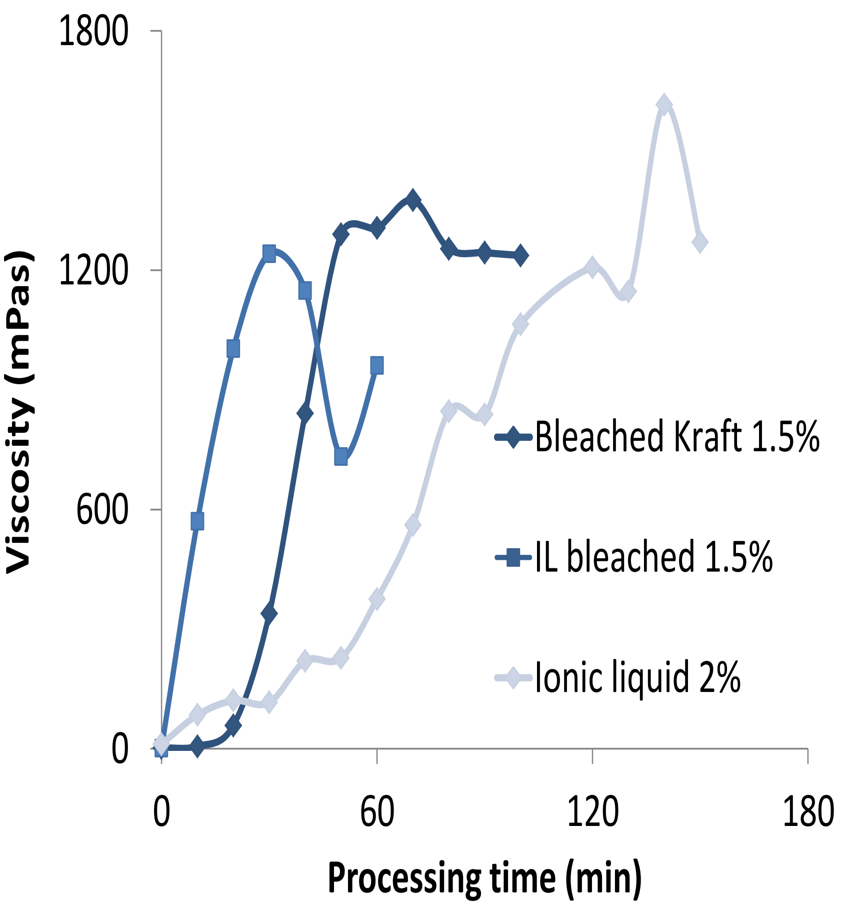
IL bleached

3-19 nm

Microstructure and viscosity

The figures displays the microstructure and viscosity measurements taken during the grinding process





Conclusions

- Cellulose nanofibers were successfully separated from all materials tested
- High elastic modulus is observed for the IL bleached birch.
- The SIL treated materials require **significantly** less mechanical processing.
- SIL treated wood materials are less energy demanding to process compared to bleached Kraft pulp.

Sample industrial collaboration: SP Processum Biorefinery Initiative and associated companies (Örnsköldvik, Sweden)

Many papers in good journals, multiple conference presentations, patent applications …

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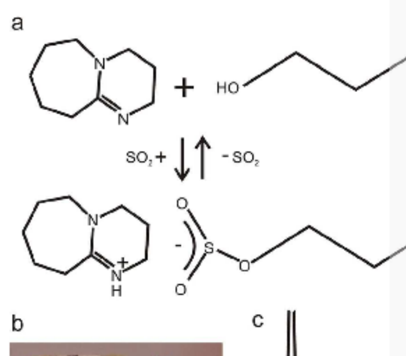
CHEMSUSCHEM Communications

Realtime ^{31}P NMR Investigation on the Catalytic Behavior of the Enzyme Adenylate kinase in the Matrix of a Switchable Ionic Liquid

Per Rogne,[a] Tobias Sparrman,[a] Ikenna Anugwom,[a] Jyri-Pekka Mikkola,[b] and Magnus Wolf-Watz*[a]

The integration of highly efficient enzymatic catalysis with the solvation properties of ionic liquids for an environmentally friendly and efficient use of raw materials such as wood requires fundamental knowledge about the influence of relevant ionic liquids on enzymes. Switchable ionic liquids (SIL) are promising candidates for implementation of enzymatic treatments of raw materials. One industrially interesting SIL is constituted by monoethanol amine (MEA) and 1,8-diazabicyclo-[5.4.0]-undec-7-ene (DBU) formed with sulfur dioxide (SO_2) as the coupling media (DBU-SO_2-MEASIL). It has the ability to solubilize the matrix of lignocellulosic biomass while leaving the cellulose backbone intact. Using a novel ^{31}P NMR-based real-time assay we show that this SIL is compatible with enzymatic catalysis because a model enzyme, adenylate kinase, retains its activity in up to at least 25 wt% of DBU-SO_2-MEASIL.



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200µm | Mag = 100 X | EHT = 15.00 kV | Aperture Size = 30.00 µm
LEO 1530 | WD = 12 mm | Signal A = SE2 | Image Pixel Size = 1.172 µm

Carbohydrate Polymers 136 (2016) 459–465

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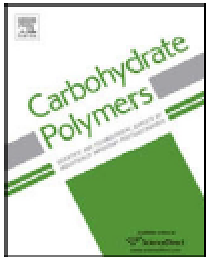

Deconstruction of Nordic hardwood in switchable ionic liquids and acylation of the dissolved cellulose

Valerie Eta[a,b,*], Jyri-Pekka Mikkola[a,b]

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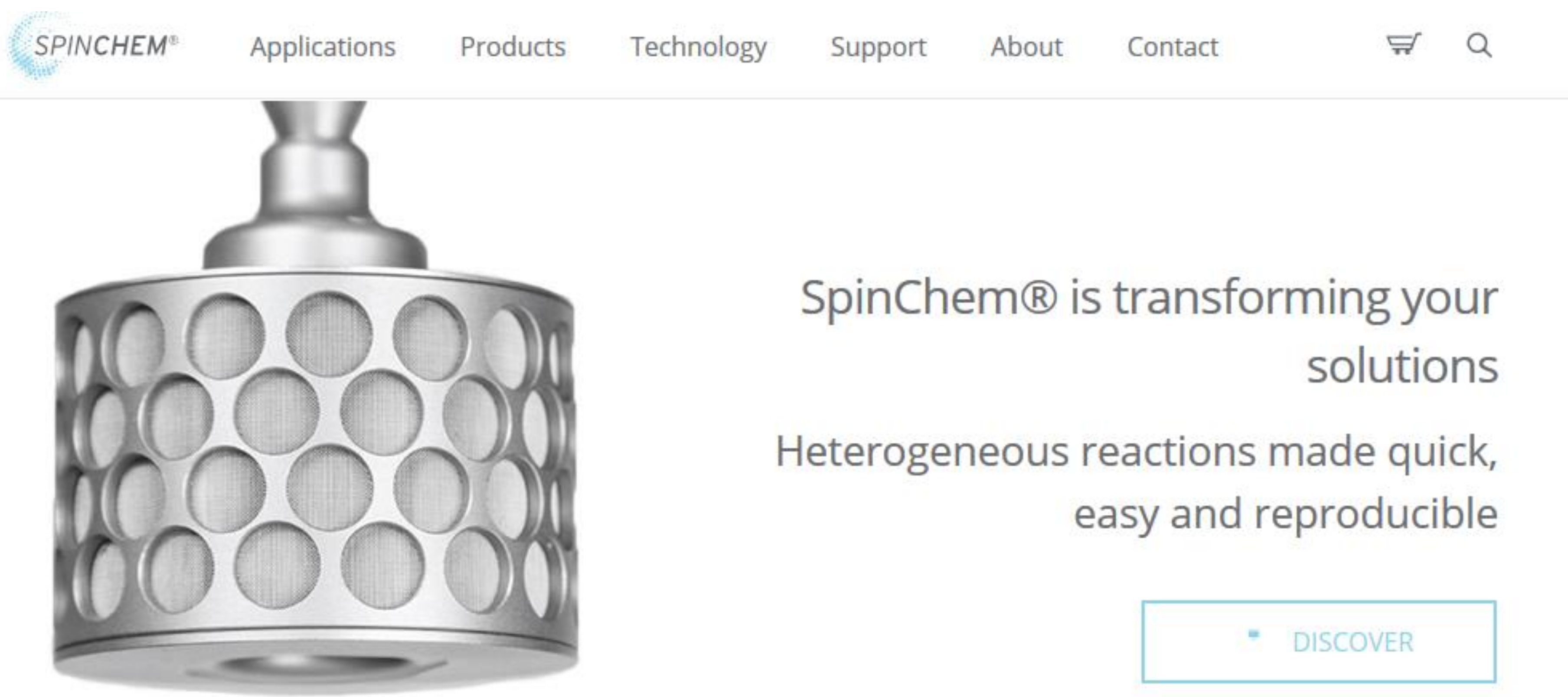
ABSTRACT

Nordic hardwood (*Betula pendula*) was fractionated in a batch autoclave equipped with a custom-made SpinChem® rotating bed reactor, at 120 °C using CO_2 and CS_2-based switchable ionic liquids systems. Analyses of the non-dissolved wood after treatment showed that 64 wt% of hemicelluloses and 70 wt% of lignin were removed from the native wood. Long processing periods or successive short-time treatments using fresh SILs further decreased the amount of hemicelluloses and lignin in the non-dissolved fraction to 12 and 15 wt%, respectively. The cellulose-rich fraction was partially dissolved in an organic superbase and an ionic liquid system for further derivatization. Homogeneous acylation of the dissolved cellulose ... ith variable degree of substitution ... n conditions, the cellulose acetate

…and this was possible because of a spin-off company from the university, www.spinchem.com



V. Eta et al. / Chemical Engineering Journal (2013)

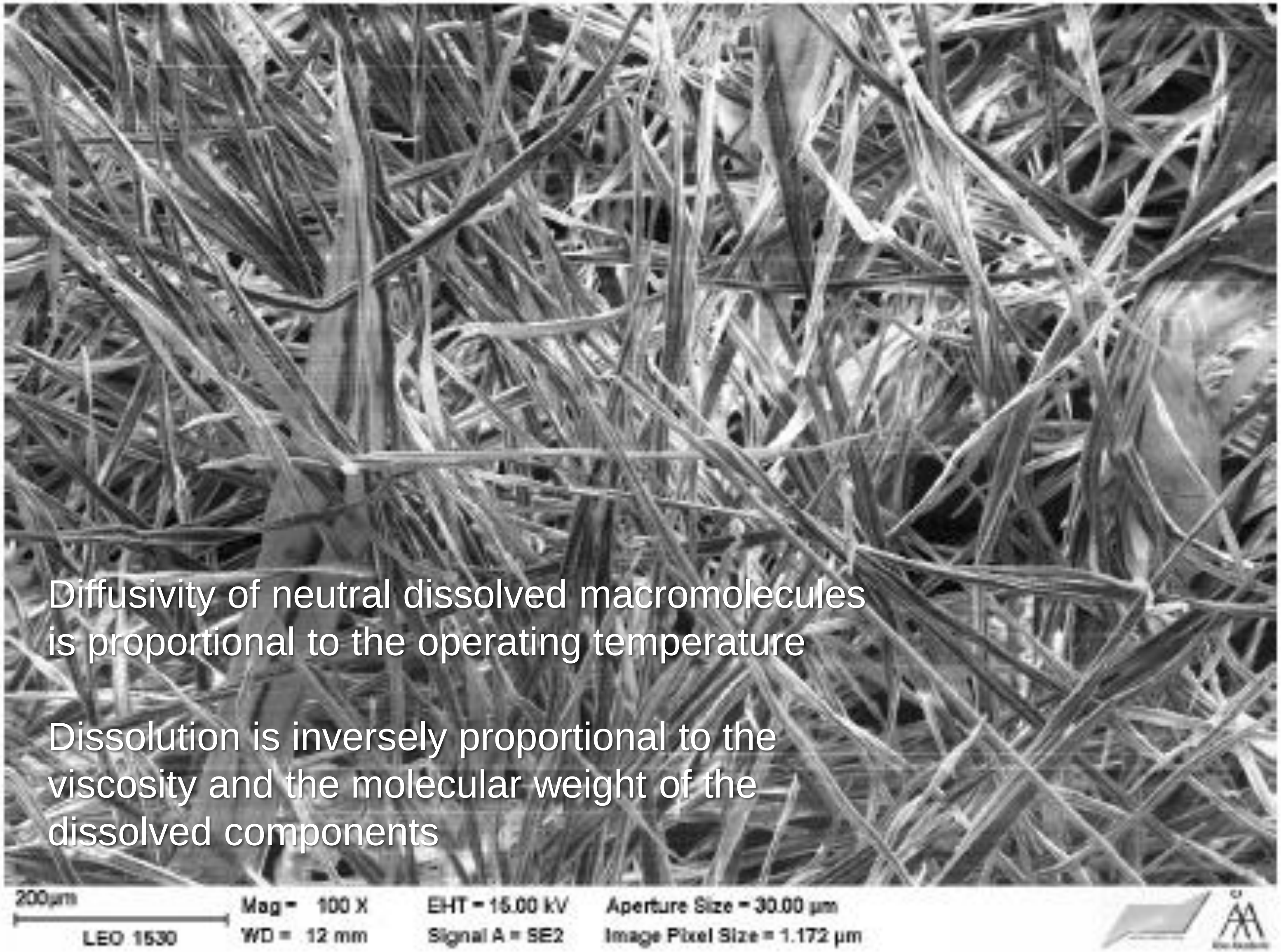


Fig. 6. SEM image of SIL treated wood chip (*B. pendula*) demonstrating the effect of SIL treatment and revealing an astonishing three dimensional fibre morphology present in Nordic wood.

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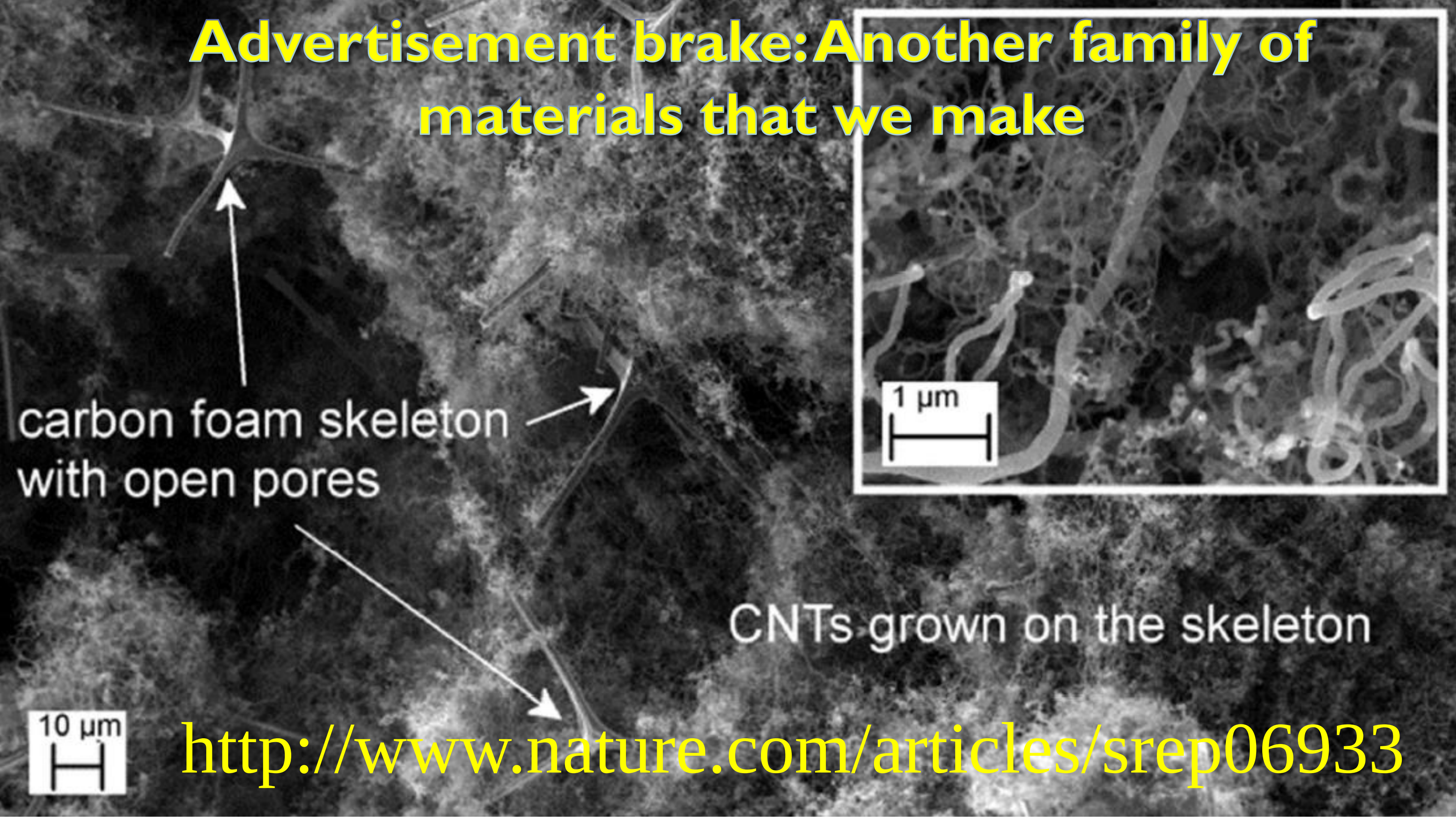

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