Cost-effective and sustainable Bio-Renewable Indoor Materials with high potential customization and creative design in Energy Efficient buildings

NCC EXTRACTION AND PROCESSING

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DESCRIPTION

This process includes an optional pretreatment steps (with acid and base), depending on the cellulose source, to remove $CaCO_3$ and hemicellulose from the source material. This step is followed by hydrolysis using sulfuric acid at high concentration. During the hydrolysis, the amorphous regions of the cellulose fibers are hydrolysed and the crystalline regions remain in the suspension. Following the hydrolysis, a separation and washing steps take place in order to separate the acid and the dissolved carbohydrates from the NCC. Finally, NCC is neutralized by titration with NaOH in order to allow a better dispersion in water as well as better stability of the material over time. The material can be stored after the titration step or it can first be dispersed in water using sonication.

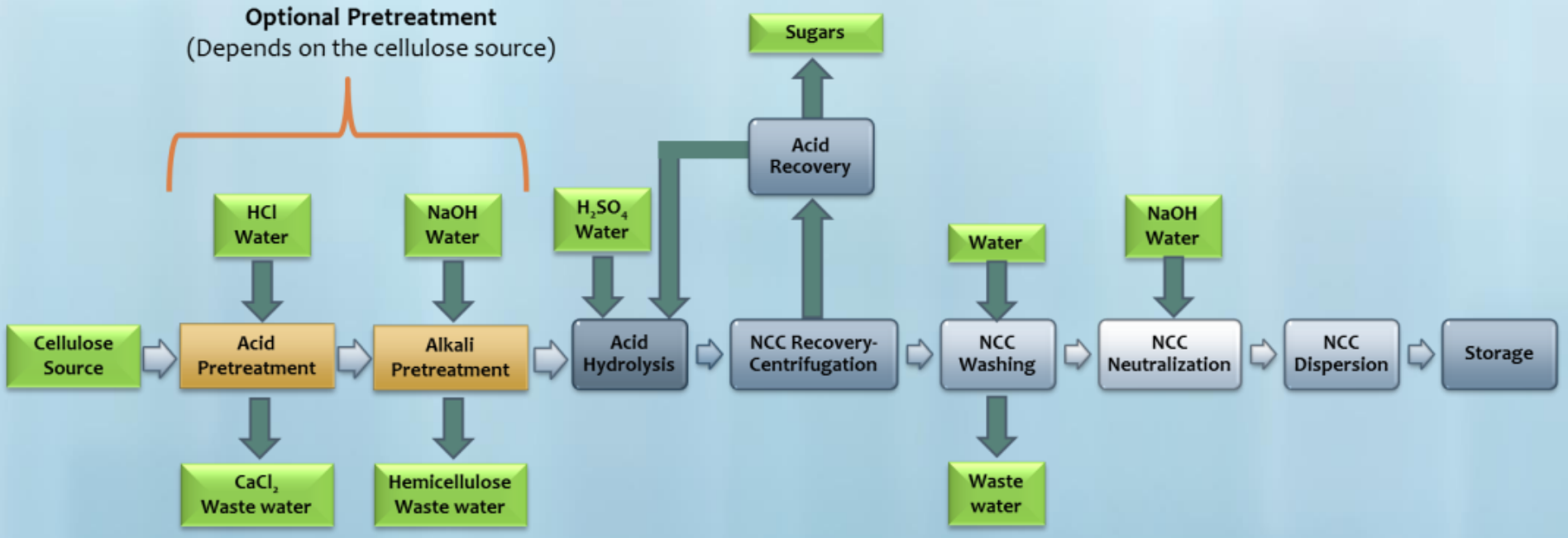
REQUIREMENTS

- For pretreatment, raw material without lignin, known concentrations of CaCO3 and hemicellulose, to adjust the concentrations of acid and base.
- Stirrer torque should be high enough to overcome the reaction overload at the beginning of the pretreatment and hydrolysis.
- Sufficient capabilities of centrifugation in terms of velocity and acid compatibility.
- QA analysis for sonicated NCC (viscosity, surface charge, Zeta-potential).

END USERS

Customers for the NCC extraction process can be either internal to the project (SILCART) using it to foam production, or any foam producers external to the project, operating it under license from the process owners.

BLOCK FLOW DIAGRAM



GUIDELINES FOR NCC EXTRACTION AND PROCESSING

Step 1 - Pretreatment

The pretreatment process is optional, based on the cellulosic source

- **Step 1a:** Acid pretreatment. In this process HCl is added to the reactor, followed by the addition of waste pulp, while stirring. During the reaction with the HCl, the waste is cleaned of CaCO3 and CO2 is released. The pulp is washed with water at the end of this process and filtered in an industrial Buchner filter system.
- **Step 1b:** Alkali pretreatment. The second step of the pretreatment involves reaction of the pulp with NaOH. NaOH is added to the reactor followed by the addition of the HCl treated pulp. In this process hemicellulose is removed. The pulp is rinsed with DW after this process and filtered in an industrial Buchner filter system.

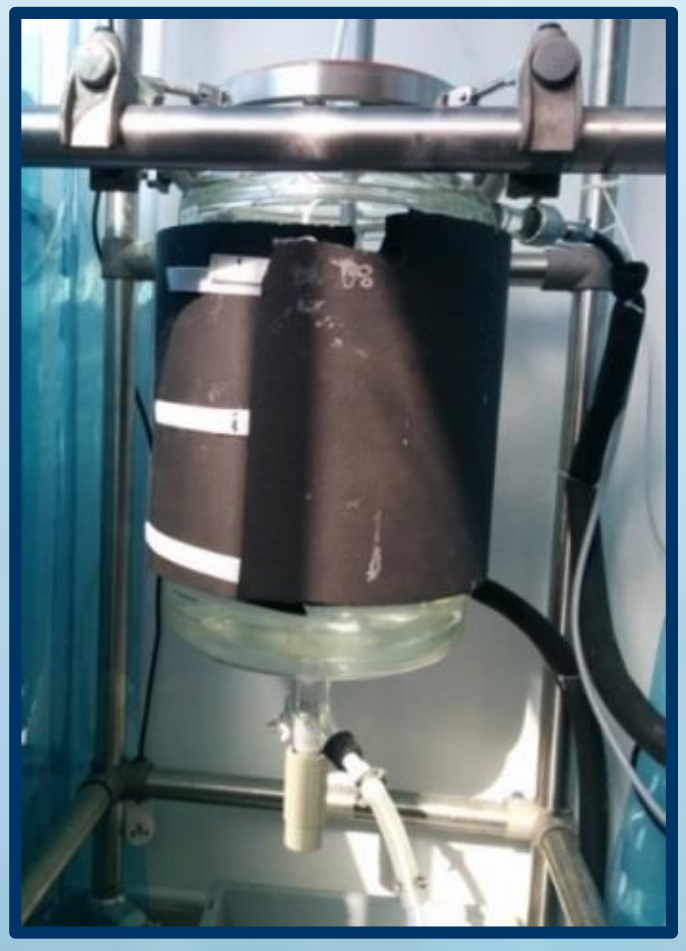
Comments

1a
- Adjust HCl concentration, according to CaCO3 concentration
- Wash the pretreated cellulose source (without CaCO3, no more CO2 released) thoroughly until reaching pH 7

1b
- Adjust NaOH concentration, according to the hemicellulose content in the given row material
- Wash the pretreated cellulose source until pH 7

Step 2 - Hydrolysis

In this step, pretreated cellulose pulp is hydrolysed with sulfuric acid. Sulfuric acid at high concentration is added to the reactor and the temperature is set to the reaction temperature. While stirring, cellulose pulp is added to the reactor. During the reaction, amorphous regions along the cellulose fibrils are hydrolysed and crystalline regions (NCC) are left in the suspension. At the end of this process, the reactor is cooled and the acid suspension, containing hydrolysed pulp and NCC, is collected and it is ready for separation.



Comments

- Adjust acid concentration, acid / pulp ratio, temperature, and reaction time according to the raw material
- During the hydrolysis, monitor rpm of stirring
- Perform QC analysis for the process

Step 3 - NCC recovery – centrifugation and cleaning

The hydrolysed cellulose is separated by centrifugation. The supernatant, containing concentrated sulfuric acid and carbohydrates, is transferred to the acid recovery process, while the pellet, containing the NCC, is redispersed in water. Further centrifugation and washing steps are done until almost all the sulfuric acid is removed from the NCC pellet.

Comments

- Check Supernatant / Pellet ratio
- Check pH after each wash

Step 4 - NCC neutralization

The clean, acidic NCC is diluted in water and titrated to neutral pH using NaOH. This allows the NCC to be better dispersed and stored.

Comments

- Adjust pH using NaOH while stirring the suspension

Step 5 - NCC dispersion

After neutralization, NCC is dispersed in water, in a desired concentration, and it is sonicated with an industrial continuance sonication system (UIP500hd, Hielscher, Germany). The sonication separates the NCC particles inside the water and result in a stable NCC suspension. At this stage NCC suspension is stored at 4 °C.

Comments

- Adjust NCC concentration to the optimal concentration for foam formulations.
- Perform sonication to obtain a transparent material.
- Make sure that the product is within the range of desired quality, by QA analysis.

GLOSSARY

- **Pretreatment**. Abstraction of any component in row material besides α-cellulose to a sufficient level that allows high quality NCC production
- **Hydrolysis**. The chemical breakdown of a β-1,4-glycosidic bonds of α-cellulose chains in the presence of acid and water. Controlled hydrolysis removes the amorphous regions of the cellulose chains.
- **NCC recovery.** The process of cleaning and retrieving not hydrolysed, crystalline cellulose particles

TROUBLESHOOTING TABLE

PROBLEMS	POSSIBLE CAUSES	EFFECTS
Pulp is not fully washed from HCl	Poor mixing, vessel not large enough for homogeneity	In step 1b, some of the NaOH will react with HCl leading to lower concentration of NaOH
Low NaOH conentration	Excess of HCl after step 1a, unknown hemicellulose content	Hemicellulose leftovers will lead to high viscosity, low transparency product, due to only partial hydrolysis of α-cellulose
High concentration of NaOH	Unknown, too low, hemicellulose content	Low yield of NCC due to alkali hydrolysis, low viscosity of product
Over-hydrolysis	One or more of the conditions occurred: exceeding temperature, duration, acid concentration, acid/pulp ratio	Low yield, possibly no NCC recovery, low viscosity
Insufficient hydrolysis	One or more of the conditions occurred: insufficient temperature, duration, acid concentration, acid/pulp ratio	Partial hydrolysis, poor quality of NCC product.
Ineffective separation	Not efficient centrifugation velocity, hydrolysis conditions were not optimal	Low yield
Insufficient washing	Not enough water used, not enough washing cycles	Excess of NaOH is required to neutralize, leading to high viscosity NCC with low transparency
Excess of washes	To many washing cycles	Loss of yield
Final pH above 8	Too much NaOH added	Can lead to poor quality NCC, up to aggregation and sedimentation, and even unusable NCC.
Too low viscosity	Over-hydrolysis or exceeding concentration of NaOH	Rheology properties not sufficient for foam production

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