

# CNC surface modification

Synthesis of CNC-Sulfonamide

Bachelor's thesis in chemistry and chemical engineering

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## **Abstract**

There are several goals in this project. The main goal is to devise a new method of synthesizing CNC-sulfonamide. Several different methods were used in order to try to synthesize CNC-sulfonamide. Some were promising but did not seem to be effective. However one method seems to have yielded CNC-sulfonamide in the end product. Unfortunately other undesired products were also present like TBAB. Still the main goal was accomplished. Secondary goals of this project were to find out at what temperature you could synthesize CNC and if all types of amines could be used to synthesize CNC-sulfonamide and those questions were somewhat answered.

## **Acknowledgment**

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## **Table of contents**

<b>1. Introduction</b>	<b>4</b>
<b>1.1 Objective</b>	<b>4</b>
<b>2.1 CNC</b>	<b>4</b>
2.2 Modifying CNC	5
2.3 Sulfonamides	5
2.4 Amines	5
2.5 NMR	5
2.6 FT-IR	5
2.7 Titration	6
2.8 Moisture Analyzer	6
<b>3. Method</b>	<b>7</b>
<b>3.1 experimental section</b>	<b>7</b>
3.11 preparing the sulfuric acid	7
3.12 Preparing the CNC	7
<b>3.2 Experimenting the amines</b>	<b>8</b>
<b>3.3 Synthesizing CNC-Sulfonamides</b>	<b>9</b>
<b>3.4 Synthesizing CNC with Malononitrile and Carbonates</b>	<b>10</b>
<b>3.5 CNC -sulfonamid alternative experiment</b>	<b>11</b>
<b>4. Results</b>	<b>11</b>
4.1 TLC	11
4.2 NMR	13
<b>5. Discussion</b>	<b>13</b>
<b>6. Conclusion</b>	<b>14</b>
<b>7. References</b>	<b>14</b>
<b>8. Appendix</b>	<b>16</b>

# 1. Introduction

In the past decade, it has become increasingly clear that the world needs to shift from the usage of fossil-based raw material to more green raw materials such as cellulose, lignin and hemicellulose from annuals and trees. [1] The problem however is that biological raw material streams contain a large amount of oxygen atoms, which makes them sensitive to moisture, another major challenge in this conversion is to modify the biological raw materials so that they become meltable and thus can be used in extruders and compression molds to produce products. A product that has become very important in that respect is Nanocrystalline cellulose. Nanocrystalline cellulose has strength and durability comparable to steel. In theory it could mean that if nanocrystals are attached in a good way then there is the possibility that you have a biological material that is stronger than steel products.

## 1.1 Objective

In this project, a chemical surface modification of nanocrystalline cellulose will be developed with the ambition of finding a chemical modification that produces CNC-Sulfonamides. Some intermediate goals have been set for this project:

1. At what minimum temperature microwave radiation can be used to synthesize the products.
2. Find out purity of reaction, what molecules are left in the end product (and if any, how many of different molecules).
3. How Sulphate groups are attached to the molecule.
4. To find out which groups of amines can be used to synthesize CNC-amides and CNC-sulfonamides.
5. To develop a new method to synthesize CNC-Sulfonamides

# 2. Background and Theory

Here some necessary background and theoretical information is given regarding CNC, modifying it and other products.

## 2.1 CNC

Nanocrystalline cellulose is a renewable nanomaterial that has recently received a lot of [1] attention for its use as a biomedical material. [4] This is due to its phenomenal physical and biological properties. Some of these properties are surface chemistry, low toxicity, biodegradability and biocompatibility. Nanocrystalline cellulose-based biomedical materials

are widely used in medical implants, wound healing, drug delivery systems and cardiovascular disease.

## 2.2 Modifying CNC

Surface modification of cellulose nanocrystals (CNC) is very important to explore new applications for these CNC. [5] In this study CNC is modified to create CNC-sulfonamide which can be used in medicine.

## 2.3 Sulfonamides

Sulfonamides are a select group of chemical compounds with antibiotic function[2]. Sulfonamides generally have the formula  $RSO_2NH_2$  with R being an organic compound. Sulfonamides are very difficult to cleave and are very rigid making them usually crystalline. Sulfonamides are commonly used in medicine.

## 2.4 Amines

Amines are groups that contain a basic nitrogen atom with a lone pair of valence electrons. [11] There are several different kinds of amines for example primary amines like Octylamine, Hexylamine and 2-ethyl-hexylamine, secondary amines like diisobutylamine, diethylamine and Di-n-octylamine, tertiary amines like n-n-Dimethylamine and cyclic amines like Benzylamine and Morpholine.

## 2.5 NMR

NMR (Nuclear Magnetic Resonance spectroscopy) is a method used in chemistry to determine the content and purity of a sample as well as its molecular structure. [14] NMR can be used to analyze mixtures containing known compounds. As for new or unknown compounds, NMR can either be used to see if there is a potential match with the spectral libraries or to infer the basic structure directly. After the structure of a compound is known NMR can also be used to determine molecular conformation in solution as well as studying physical properties at the molecular level such as conformational exchange, phase changes, solubility, and diffusion. NMR is usually used to analyze either groups of protons in a compound or carbon 13 groups.

## 2.6 FT-IR

FTIR or Fourier Transform Infrared Spectroscopy is an analytical technique used to identify different organic compounds.[15] In FTIR spectroscopy, a sample (solid, liquid or gas) is irradiated with an

infrared light beam of several frequencies and the amount of absorbed light is measured. The information obtained (the absorption at each wavelength) is unique depending on the chemical composition of the sample, which enables the identification of, for example, a previously unknown substance. Typical uses are to identify:

- Organic substances such as polymers, oils or fats
- Inorganic substances such as minerals.
- Functional groups such as alcohols, esters amines etc.

## 2.7 Titration

Titration is a common method of analysis in chemistry used to determine the concentration of a solution, by reacting it with a solution of known concentration. [16] A solution of known concentration (the titrator) reacts with another solution, which has an unknown concentration. The titrator solution is added a little at a time. The reaction is such that it is possible to detect when a certain molar ratio between the solutions has arisen, for example, the combined solution changing color. Then you stop adding titrators. By adding the titrator from a burette, you can subsequently read how large a volume of titrator solution has been added. Using knowledge of the two volumes and the molar ratio that will apply to the titration reaction, one can then calculate the concentration of the solution. Acid-base titrations depend on the neutralization between an acid and a base when mixed in solution. The acid-base indicator indicates the endpoint of the titration by changing color.

## 2.8 Moisture Analyzer

A Moisture Analyser, is an instrument used for the determination of the moisture content of a sample. It consists of an infrared weighing and heating unit. [17] Moisture analyzer is used in several analyzes / industries and therefore it is an extremely important analysis. Examples of areas where the instrument is used are the food-, pharmaceutical- and agricultural industries. This affects quality control criteria such as weight, durability and quality.

Most products on the market must maintain an optimal moisture content for the highest quality. If there are high levels of moisture in the product, both the value but also the quality of the product decreases. This is because there is a greater potential for microbial activity. But there are also laws and regulations that determine the correct moisture content for certain regulated products. An example is national food regulations that define the maximum allowable moisture content for certain food products.

## 3. Methods

The project focuses on both theoretical analysis and experimental parts. In order to get a proper understanding of the project a literature analysis was made on specific words related to the project like Nanocrystalline cellulose, sulfonamide and Surface modification of cellulose nanocrystals (CNC). After the literature analysis experiments were performed and will be described below.

### 3.1 experimental section

#### 3.11 preparing the sulfuric acid

The first step is to synthesize a 64% sulfuric acid about two days before the hydrolysis.[3] This is done by first pouring 720 ml of deionised water in a container. After which the stirring is started at about 120 rpm. It is important to make sure that the stirrer does not go against the stirring anywhere and after which a thermometer was put in. It is also important to make sure that the thermometer does not get hit by the propeller blades. When all that is mentioned before is completed, pour 700 ml of 99% sulfuric acid in a separate 2 liter container. Drop speed should not exceed 1 drop per second because of the fact that adding sulfuric acid to water causes a very exothermic reaction. After all of the acid has been poured in, the reaction was allowed to ‘mature’ for one day before the cellulosa was added. The heating started in the afternoon on the day before hydrolysis. The temperature was put at 60°C and that makes it 45°C in the container. It is also important to make sure that there are no air holes in the tubes.

#### 3.12 Preparing the CNC

First the temperature of the sulfuric acid was checked. [3]After which the speed of the mixture was increased to about 175 rpm and 400 grams of Avicel was carefully and slowly poured in carefully and slowly using a spoon so that no lumps would form. When everything was poured in and no big lumps could be seen the speed was decreased to about 150 rpm and the reaction was allowed to stir for about 2 hours. During the wait 8 dialysis buckets were cleaned and prepared. When almost 2 hours had gone the reaction was poured in a bucket with 7 liters of deionised water. After which the reaction was put in 8 dialysis buckets which were then centrifuged 4 at a time for 15 minutes. When those 15 minutes were done the clear liquid was poured out, the CNC dispersed in deionized water and they were centrifuged again. This was done until some of the CNC was in the clear liquid which made it hard to pour it out. [4] When that happened the CNC was put in a dialysis membrane and then in deionised water. The water was changed only a few minutes after the CNC in the dialysis

membrane was initially put in. After that the CNC was left alone in the water until the next day. In the morning between 8-9:30 and in the afternoons between 15-16:30 every afternoon the conductivity was measured and the water was changed. This was done for days until the conductivity was below 5 us. When that happened the CNC was left alone in the water without having to change. If the conductivity remained below 5 us for 3-4 days then it was finished. When that happened the CNC was put in a bucket and stirred to remove all the clumps After which the dry content was measured and in this case appeared to be about 2.8 wt%.

## 3.2 Experimenting the amines

While the water was being changed, an experiment was performed with another CNC-SO<sub>3</sub>H in water that had a concentration of about 4.5%. [8] First, a small portion of CNC-SO<sub>3</sub>H was taken to measure the dry content and to make IR. Then a large part of the substance was taken and the solvent was changed to acetone (other solvents such as THF and acetonitrile had also worked). This was done by adding 20 grams of CNC-SO<sub>3</sub>H in water to about 300 ml of acetone. After that, CNC-SO<sub>3</sub>H was dissolved to separate from the acetone. A small portion of the solution was taken to measure the dry content and another for IR 0.09 grams of 2,4,6 trichloro-1,3,5-triazane in 2 ml of acetone. Then that solution was added by CNC together with 4 ml of acetone. After that, 0.07 grams of triethylamine was dissolved in 1 ml of acetone and then it was added with the other substances. Then the solution was placed in a microwave oven with temperature rise up to 80 degrees in 20 minutes and then it was cooled down to 30 degrees to get the substance CNC-SO<sub>2</sub>Cl. When ready, the reaction was filtered and washed with acetone. The dry matter content was then measured and a small portion of it was also taken for IR.

Then 3.1 grams of sulfonyl chloride was dissolved in 4 ml of THF and mixed with CNC-SO<sub>2</sub>Cl. 0.08 grams of benzylamine was also dissolved in 1 ml of THF and mixed with the solution. Then 4 ml of THF was added to the solution before the solution was put back into the microwave oven and heated to 50 degrees in 10 minutes and cooled to 30 degrees. When ready, material was filtered and washed with acetone. The new material was labelled CNC-SO<sub>2</sub>B6 The dry content was measured and a small portion was taken for IR. The experiment was repeated and 4 different vials were made with two different amines. Benzylamine from the first experiment was dissolved in THF put in two vials of CNC-SO<sub>2</sub>CL with 4 ml THF and Hexanamine was also dissolved in 1 ml THF and put in the other two vials of CNC-SO<sub>2</sub>CL with 4 ml THF. One of each of the vials was put in 80 degrees for 20 minutes in the microwave while the other two were put in 60 degrees for 20 minutes to test if the experiment could be done in lower temperatures. After which a sample of each vial was taken for IR.



### 3.3 Synthesizing CNC-Sulfonamides

A sample of 5.2 grams of CNC was first taken and stirred in water . [18]After which 0.2 ml of Diethylamine and 0.2 grams of Iodide was added. [10] A few drops of Sodium hydroxide were added. The reaction was stirred for 4 hours.[9] Then a sample was taken, dried in an oven and later used for IR. The experiment proved to be a success.

In order to confirm that the experiment works a sample was made without CNC. [6] 0.38 grams of Toluene-4-sulfonic acid was weighed and stirred with water. After which 175 mg Diethylamine, 0.253 grams of Iodide was added and 2-4 drops of sodium hydroxide were also added. Then the reaction was allowed to stir for 4 hours in a dark environment. When it was finished the reaction was put in a separation funnel and DCM along with Sodium metabisulfite was added to cause the separation and get rid of the iodide and the whole funnel was stirred. When the reaction had settled into two different liquid phases the upper half was put in an evaporator. When all of the DCM solvent was gone an NMR, TGA and an IR was done. However in order to purify the product a chromatography was done with the 3 vials being the product. The results were promising and can be seen in results.

Due to the success of the first experiment another attempt was made using 0.38 grams of Toluene-4-sulfonamide again. However this time Di-n-hexylamine was used instead. Approximately 0.557 ml of Di-n-hexyleamine was in the reaction along with 0.253 grams of Iodide and 2-4 drops of Sodium hydroxide. The reaction was left to stir for 4 hours and after that samples were prepared for NMR. Then the remaining product was put in a separation funnel and DCM along with sodium metabisulfite were added. Just like before the upper half of the liquid phase was put in an evaporator and then a small sample was used for IR and NMR. Afterwards the remaining sample was centrifuged with deionised water in order to purify it. When that was finished a sample was taken for FTIR.

In order to confirm that the experiment is a success it was expanded upon. Just like before 0.38 grams of Toluene-4-sulfonic acid was weighed. In the reaction along with 0.253 grams of Iodide. However 7 different amides were used along with 2-4 drops of NaOH each time. The amines can be seen in the table below.

Amines	Volume ( ml)
Octylamine	0.4
Benzylamine	0.265
2-ethyl-hexylamine	0.39
Di-n-octylamine	0.726

diisobutylamine	0.42
n-n-Dimethylamine	0.24
Morpholine	0.21

In each reaction 2-4 drops of NaOH were also added. After which it was stirred for 4 hours in a dark environment (covered by aluminium). After Which the product was put in a separation funnel. DCM and Sodium metabisulfite were added. The upper half of the liquid phase was put in an evaporator and then a small sample was used for FTIR and NMR.

Another variant of the experiment was made using 0.38 grams of Toluene-4-sulfonic acid again. 0.253 grams of Iodide, 0.4 ml of octylamine and 0.16 ml of pyridine was used instead of NaOH. After which it was stirred for 4 hours in a dark environment ( covered by aluminium). Just like the previous experiment the remaining product was put in a separation funnel and DCM along with Sodium metabisulfite was added. Again the upper half of the liquid phase was put in an evaporator and then a small sample was used for FTIR and NMR.

### 3.4 Synthesizing CNC with Malononitrile and Carbonates

First 5.0104 grams of CNC with a molar concentration of about 300 umol was measured. This was stirred in deionised water. Afterwiche in a separate beaker 0.126 grams of Potassium Hydrogen carbonate and 0.01 grams of Malononitrile were stirred in a beaker with Diionised water for 20 minutes. Whan that was done It was put in with the CNV and was allowed to stir for 15 hours. After Which more water was added and it was centrifuged 3 times with water and 3 more times with water and 5 grams of ethanol in order to purify it. Then a sample was taken for Ir , TGA and another for NMR. The experiment however proved to be a failure,

Another variant of this experiment was made. 20 grams of CNC was put in two different beakers.

#### **Beaker 1**

In the beaker 0.3 ml on NaOH and 0.076 grams of Iodide were added.

#### **Beaker 2**

In the beaker only 0.024 ml of pyridine was added.

Both beakers were allowed to stir for 2 hours. 1 hour into the stirring 0.08 grams of Malononitrile and 0.38 grams of Sodium Carbonate were measured and put together to stir in a third beaker for 0.5-1 hour. After Which half of the mixture in the third beaker was added into beaker 1 and the other half in beaker 2. Then both beakers stirred for an additional 2 hours. When the stirring was finished both of the products in the beakers were centrifuged

using 50 ml deionized water with 5 grams of ethanol. Then a sample was taken of each beaker for FTIR and NMR.

## 3.5 CNC -sulfonamid alternative experiment

A new attempt to synthesize CNC-sulfonamide was made. [7] Two simultaneous experiments were made. In the first solution 10 mmol ( 1.2212 grams) of benzoic acid is poured in saturated in 50 ml sodium bicarbonate. In the second solution 10 moles of para-toluenesulfonic acid were poured in another 50 ml saturated sodium bicarbonate. The solutions were both cooled down to about 0 degrees celsius and kept that way. Then 1 mmols of tetrabutylammonium bromide was allowed to stir for 30 minutes. After Which the temperature was checked on again to ensure that it was 0 °C than 12-13 mmol of TCT ( Cyanuric chloride) were dropped slowly into both solutions. The solutions were then allowed to stir for another 30 minutes. Afterwhich the ice used to cool the solutions was removed and 1.653 ml of hexylamine was poured in each solution. After Which it stirred for another 30 minutes. When the solution had finished stirring 20 ml DCM was added in each solution and was stirred. This was done again with another 20 ml of DCM being poured in. After Which the solutions were filtered. Then a Column chromatography was performed on the experiments. The solvent used was 90% DCM and 10% ethanol. After 13 vials 20% methanol and 80% DCM was used. And after confirming on a tlc plate which of the vials had a product the DCM vials were evaporated using a evaporator. Then an nmr was used to see what products were left in the round beakers.

## 4. Results

### 4.1 TLC

**Far Left=Benzoic acid**

**Left= Tuolo sulfonic acid**

**Middle left= tetrabutylammonium bromide**

**Middle right= Hexylamine**

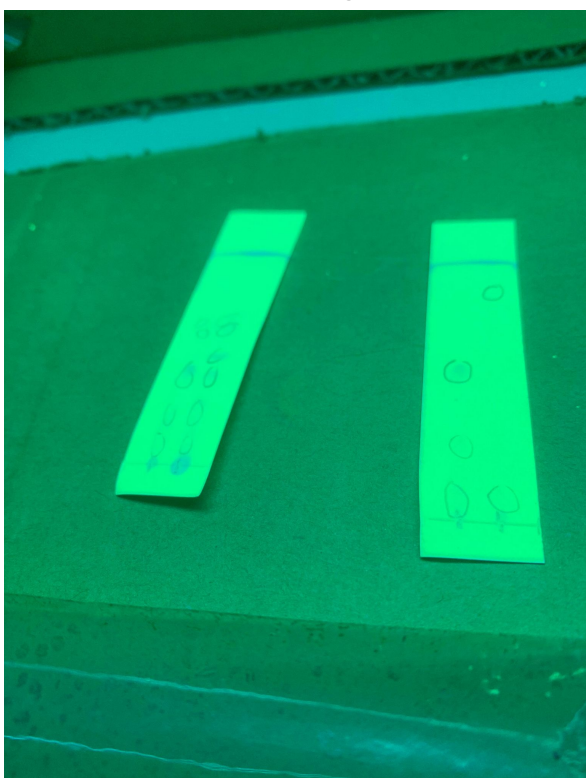
**Right=Benzoic Acid-hexylamine**

**Far Right= Toulol sulfonic acid-hexyl amine**

**TLC with clearing fluid**



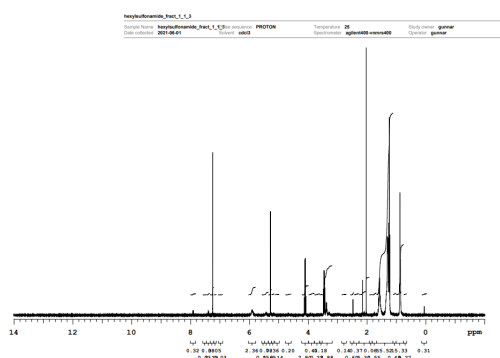
Left= Benzoic acid Right= Tuolol sulfonic acid



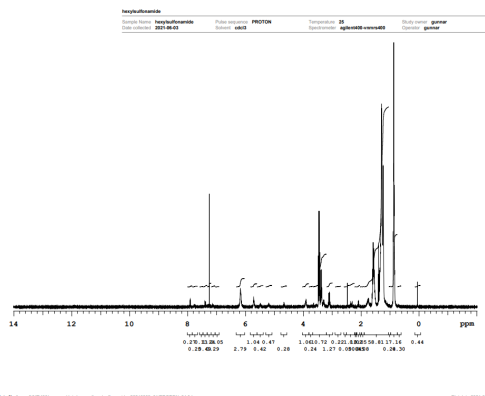
From the way marked spots in the TLC for Toulol sulfonic acid are different, it can be determined that a product has been formed and that very little if any at all of the original Toulol sulfonic acid material remains.

## 4.2 NMR

Before chromatography



After chromatography



### Toulol-sulfonicacid before and after chromatography

In the NMR hexyl amine can be observed between 0.7-1.8. It can also be determined that there is some product in the sample due to the tops between 3-4. A potential product can be found in that area but one of the tops is lilley for Toulol sulfonic acid. But the sample also contained TBAB (Tetrabutylammonium). Between 6-8 is most likely the other tops for Toulol sulfonic acid and it matches with the references[13].

All FTIR and NMRs performed along with data for the CNC conductivity can be found in the appendix. A reference [12] was used to determine if the FTIRs were correct or false.

## 5. Discussion

The TLC result looked promising. On the TLC plate, the reference (left) and the sample (right) were compared. The results showed that the Tuolol sulfonic acid was in the sample. As shown in the pictures above. Due to some dots on the TLC plate it was suspected that there was TBAB in the sample. A chromatography was done to purify the product from TBAB. But in the NMR results after the chromatography it still showed some traces of TBAB. This could be because of the solvent that was used (10% methanol, and 90% DCM). After 13 vials were filled a change was made to the solvent and 20% methanol and 80% DCM was used. Due to high concentration of methanol in the solvent, some traces of TBAB most likely ended up in the product.

Several different experiments were made in order to synthesize CNC-sulfonic amide. The first experiments were in the microwave. The experiment seemed to work but an alternative more efficient method was sought after. The first attempts failed most probably due to the use of potassium ion. The high positivity of Na<sup>+</sup> likely prevented any bond. Another attempt was made using pyridine. At first the method seemed successful but after several NMR and FTIRs

were done it was determined that it most likely did not work. The products seemed to be in the samples but they were most likely not bonded or different undesirable products that were similar were instead synthesized. Other experiments using Malononitrile on CNC were performed. However after several experiments, different modifications to the experiment like using pyridine were made and different NMR and FTIR it was determined that the reactions were not a success.

In the experiments several things occurred that could have impacted the results. One thing is that the experiments could be done more carefully and one could try to be more precise when pouring and mixing material. One thing could be that the temperature might have gone up to 4 degrees celsius at some point which also could have impacted the results. The TCT might also have been poured in too quickly which might have caused the reaction with the solution to be less effective. Another possibility is that the solvent used in chromatography could be too polar for the reaction which might have affected the NMR.

## 6. Conclusion

There were many goals in this experiment. One of the main ones was to synthesize CNC-sulfonamide and several methods were used to attempt it. However only one of the methods seems to be effective. The results from the NMR while it shows some products are present other material most likely TBAB is also present. Further research is required in order to perfect this new method for creating CNC-sulfonamide.

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## 8. Appendix

### **FTIR**

[https://drive.google.com/drive/folders/1tYWC6\\_s\\_4Gq9iAUU4a-nL9utd0tGobYw?usp=sharing](https://drive.google.com/drive/folders/1tYWC6_s_4Gq9iAUU4a-nL9utd0tGobYw?usp=sharing)

### **NMR**

<https://drive.google.com/drive/folders/1K6ATl7Hg5LJ7ewMnTwkSzvADD6PlLFXZ?usp=sharing>

### **CNC conductivity**

[https://drive.google.com/drive/folders/1nmFNpg92rvSj59kGRVAijlAlq\\_s0hHbN?usp=sharing](https://drive.google.com/drive/folders/1nmFNpg92rvSj59kGRVAijlAlq_s0hHbN?usp=sharing)