# Closing the loop: use 3D-printing to produce your own filament

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Produce (Music) instruments, mobile phone cases, clothing, toy figures, but also furniture and even houses. It's all possible with 3D-printing!

## 3D-printing

3D-printers use filament to product 3D objects. In this research (bio)plastics are used for producing filament.

## Problems

- Filament is expensive
- A very expensive commercial pulling system is needed for making your own filament

## Solution

Making your own filament: cheap self-made pulling system by using 3D printing. This system can be easily adjusted to the desired diameters by changing the speed of the extruder or driving system. This is required because different 3D printers need different diameters for the filament.

## The system

The materials are 3D-printed gears household materials, such as rubber paint rollers, holding clamps, a very cheap electric motor with a maximal of 50 rounds per minute, tape and a rubber band.

## Used conditions

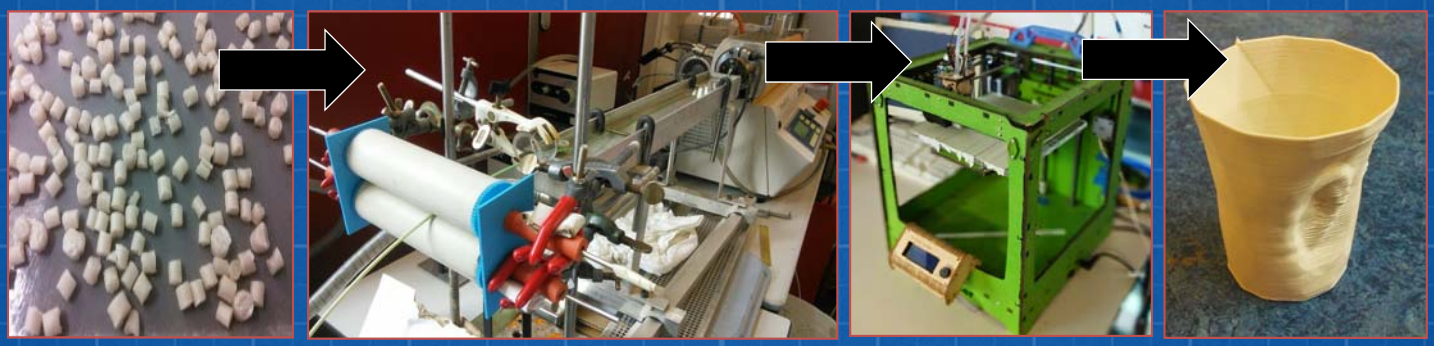
Various (bio)polymers (PLA, Bio-Flex, Solanyl and PHBV) were tested using this system for 3D printing and all of these (bio)polymers were made into an endproduct with the Ultimaker original 3D-printer.

## Results

The average deviation of this system is namely between 0.05 and 0.10 mm. The filament is round, straight and the speed is constant.

## Conclusion

- Much cheaper
- Same quality

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# Hydrogenation: A comparison between two reactors

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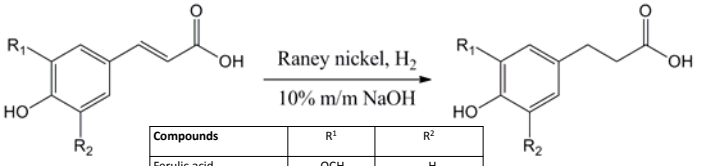
## Introduction

Because of the growing problems with non renewable plastics the past years, there is a rising interest in the production of polymers from biobased resources. Previous research performed by the Lectureship Biobased Products suggested that these polymers could be synthesised from Lignin derived compounds. A major part of the synthesis route for the production of these polymers is a hydrogenation reaction. This can easily be done on a smaller scale which gave us insight into the problems of catalyst degradation and many other factors. Eventually a large scale synthesis protocol was made that gave equally good results when compared to a commercial reactor.

## The synthesis

In order to reduce the double bond of the Lignin derived monomers, the compounds were subjected to an alkaline environment using Raney nickel as catalyst with hydrogen gas. (figure 1.)




| Compounds | $R^1$ | $R^2$ |
|---|---|---|
| Ferulic acid | - $OCH_3$ | - H |
| Sinapinic acid | - $OCH_3$ | - $OCH_3$ |

Figure 1-Reduction of Lignin based monomers.

Important aspects of this reaction are catalyst/gas dispersion and catalyst longevity. Analysis was done using HPLC.

## Self built reactor

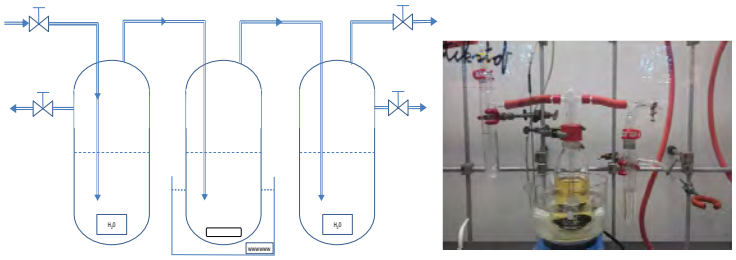


Figure 2- Self built reactor

Past versions of the reactor were not properly airtight leading to the oxidation of the catalyst, decreasing its potency (figure 3). By using two schlenk containers joined to 500mL reaction vessel (figure 2).

This new setup gave a full hydrogenation of ferulic and sinapinic acid within 8 hours at temperatures of 60-80 C°.

Isolation of the product gave a yield of 97% on a 10 gram scale.

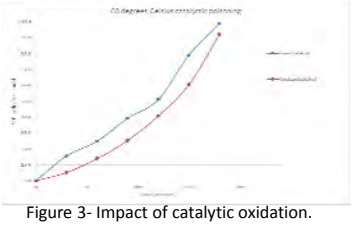


Figure 3- Impact of catalytic oxidation.

## Parr shaker reactor

The same two compounds were reduced with a Parr reactor 3910 shaker reactor in a pressurised environment (3 bar), after two hours of reaction a sample was taken for analysis. Furthermore, the same product purity and yield was attained but on a 30 gram scale.

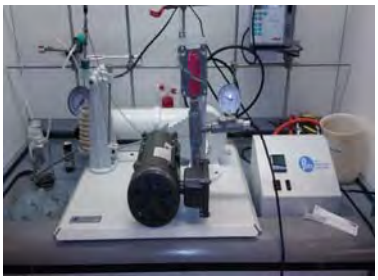


Figure 4- A Parr shaker hydrogenation reactor.

## Comparison of the results and reactors

Self built reactor:

- Full conversion of dihydroferulic acid, which was confirmed to be 99% < pure, reliably within 8 hours.
- A working reactor was built that can produce 10 grams of product per synthesis.
- The reactor was able to compete with a commercially available product.
- The reactor is cheap and simple to operate, fits perfectly for smaller lab scale reactions.

Parr reactor:

- Far shorter reaction times, due to improved gas/catalyst dispersion and increased pressure.
- Ability to work on an even larger scale.
- Has a larger container of hydrogen gas.
- Easier to control the exact reaction temperature.

## Conclusion

With the problem of catalytic degradation dealt with, the synthesis is even more economically viable because the material can be used multiple times. The final result is a reactor that boast the same product purity and isolated yield as a commercial grade reactor.

Our method proves that specialised equipment for hydrogenation synthesis is not required under mild circumstances and it is an easier model for lab scale research.

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