OPTISOCHEM

"OPTimized conversion of residual wheat straw
to bio-ISObutene for bio based CHEMicals"

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Deliverable D3.4

First report on the delivery of several tons of 2G isobutene, according to program, with full characterization

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Document Abstract

This report is the first of a series of 3 (the present D3.4, future D3.10 due for M36-May 2020 and future D3.11 due for M48-May 2021) which describes the evolving status of the WSH-based bio-IBN towards the ton scale target alongside the project life. Because of the public (PU) nature of this deliverable, details such as WSH composition, strain genotype and process parameters will not be disclosed in this document.

Various WSH samples and IBN producing strains have been tested at the lab scale and the best combinations were transferred to GBE's Pilot Plant facility at Pomacle-Bazancourt (France), for scale up. This work resulted notably in the production of the first 2G isobutene kilogram from Clariant's Wheat Straw Hydrolysate (WSH), as described in a former public report published at M12 (Deliverable D3.1). Additional kilograms were produced at the pilot plant afterwards.

For production at the ton scale, the process was transferred to GBE's Demo Plant facility in Leuna (Germany). This facility includes a fermentation unit comprising a 500L seed fermenter and a 5000L production fermenter, a purification unit allowing for IBN purification and condensation and a storage unit. Scale-up attempts at the Demo scale in Leuna with WSH started in Q4 2018.

A first series of three runs was conducted in Leuna with WSH as substrate, but several technical issues had to be overcome, thus limiting bio-IBN production to 67 kg during these 3 runs. Intense investigations have been conducted to alleviate these issues for the next production campaign, in order to achieve production rates compatible with ton-scale production.

In between, bio-IBN produced at Leuna and Pomacle was sent for analysis, and several impurities were identified, allowing to create specification sheets for bio-IBN conversion into di- or poly-isobutene. Optisochem activities dedicated to ensuring good quality for the final end-products will fine-tune the downstream processing of IBN, in order to achieve desired quality. Work is under way.

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Abbreviations

GBE: Global Bioenergies
IBN: Isobutene
WSH: Clariant's Wheat Straw Hydrolysate
TPED: Transportable Pressure Equipment Directive
PIB: Polyisobutylène ou Polyisobutène
DIB: Diisobutylène ou Diisobutène
HMF: Hydroxymethylfurfural

1. Introduction

This report is the first of a series of 3 (the present D3.4, future D3.10 due for M36-May 2020 and future D3.11 due for M48-May 2021) which describes the evolving status of the WSH-based bio-IBN towards the ton scale target alongside the project life. Because of the public (PU) nature of this deliverable, details such as WSH composition, strain genotype and process parameters will not be disclosed in this document.

GBE develops an IBN production process by fermentation, allowing for the conversion of sugars into gaseous IBN. The fermentation gas is purified and condensed, in order to recover highly concentrated IBN in pressurized cylinders. Clariant has delivered several samples of WSH to GBE in order to assess the current production process with this feedstock, and to develop new IBN producing microbes, adapted for WSH fermentation.

The adaptation process is ongoing in Evry's research facility based in Evry. The objective is to optimize the simultaneous consumption of the different sugars present in WSH, and to overcome the inhibitory effect of impurities such as HMF and furfural. Process development is first conducted at the lab scale (1 and 10 litre bioreactors) in order to test and maximize IBN production with the 1G strain as well as with its WSH-adapted counterparts.

The first conditioning of IBN from WSH were conducted in GBE's Pilot Plant facility at Pomacle-Bazancourt (France), for scale up. This work resulted notably in the production of the first 2G isobutene kilogram from WSH, as described in a former public report (Deliverable D3.1). Further activity at Pomacle, resulted into additional bio-IBN produced and bottled with WSH as a feedstock.

For production at the ton scale, the process was transferred to GBE's Demo Plant facility in Leuna (Germany). This facility includes a fermentation unit comprising a 500L seed fermenter and a 5000L production fermenter, a purification unit allowing for IBN purification and condensation and a storage unit. It is hosted and operated by the Fraunhofer CBP.

Whereas GBE's IBN production process remains today significantly less stable with WSH than with first generation sugars, it was expected both from the performances at the lab scale on WSH and from the benchmarking at the 5000L scale with first generation sugars (not shown in this public report) that the first ton could be produced rapidly. Thus, scale up attempts at the Demo scale in Leuna with WSH started in Q4 2018.

2. Production of bio-IBN in Leuna

2.1. Strain and protocol development in the Evry laboratory

Strain genotype and detailed process are confidential information. Basically, GBE's strain for IBN production have an IBN metabolic pathway, introduced into a bacterial chassis derived from the

E. coli MG1655 bacteria, and modified in order to increase yield. The metabolic pathway can convert the pivotal acetyl-CoA molecule, a central intermediate in metabolism, into IBN, through a cascade of enzymatic reactions performed by a series of recombinant enzymes. These recombinant enzymes include natural enzymes as well as artificial biocatalysts, generated by GBE's enzyme engineering platform, and catalysing non-naturally occurring reactions.

2.2. The IBN Demo Plant

GBE has constructed and commissioned a 500 litre Pilot Plant in 2014. This plant was designed for a production capacity of 10t of IBN per year. The plant including a fermentation unit and a purification unit was described in previous reports (Deliverable D3.1).

A highly automated Demo Plant was constructed and commissioned in 2016 in Leuna near Leipzig, Germany (see Figure 1). Designed for a production capacity of 100t of IBN per year, this facility includes a fermentation unit comprising a 500L seed fermenter and a 5000L production fermenter, a purification unit allowing for IBN purification and condensation and a storage unit (Figure 2). It is hosted and operated by the Fraunhofer CBP.

Figure 1. The 5000 litres IBN demo-plant in Leuna (Germany).

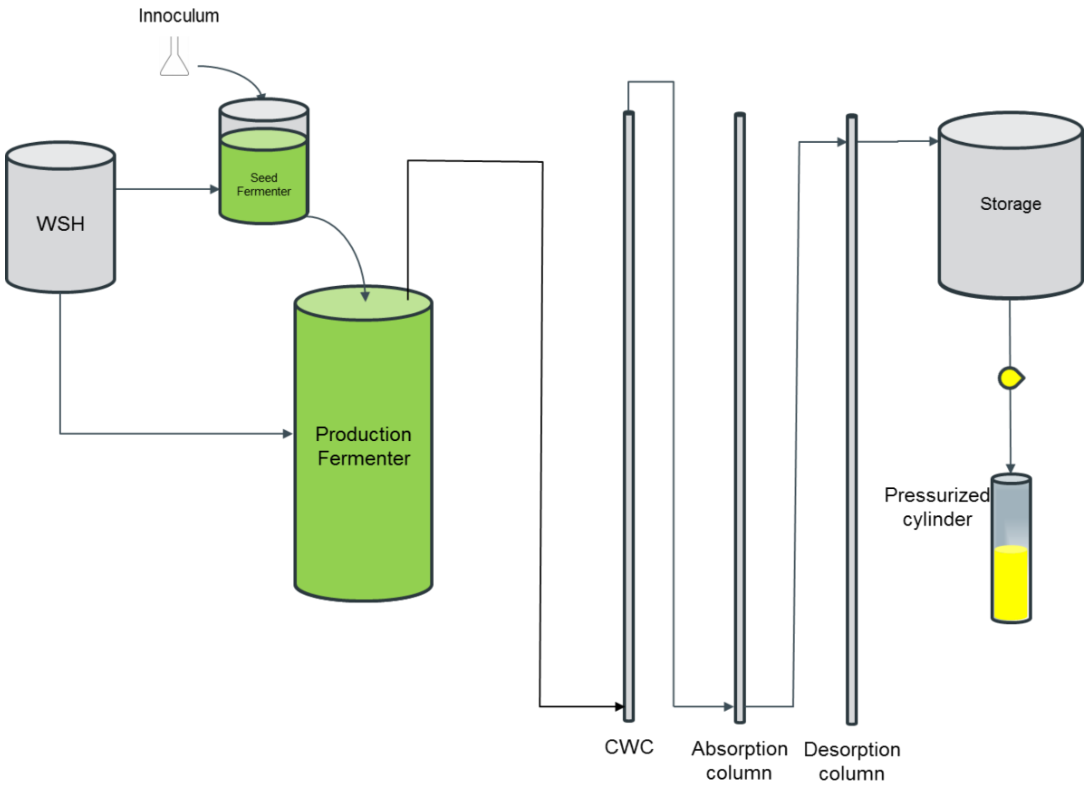

Figure 2. Process scheme of the IBN demo-plant.

The plant includes a fermentation and a purification unit as described in Figure 2. Along are present a Cleaning-In-Place (CIP) equipment as well as the pre and post treatment (i.e. HTST, surge and killing tank, centrifuge) equipment. The fermentation unit comprises a Seed fermenter and a Production fermenter, a series of satellites tanks for substrate, additives, basic, acids and cleaning solutions. The purification unit comprises a washing column (CWC), adsorption and desorption columns and a final storage and product recovery system

2.3. Bold lines of the IBN production process

As for the smaller scales, pre-cultures are prepared in Erlenmeyer flasks, in parallel, the clean fermenter is prepared with fermentation medium and sterilized, instrumentation (pH, DO, gas analysis) is calibrated, and safety systems are checked.

Upon inoculation, the growth phase is conducted in a fed-batch mode, in order to reach the desired biomass concentration and physiological state. When the cell density reaches a certain level, the broth is transferred to the Production fermenter in order to finish the growth phase until the plateau. At this stage, the production phase is initiated, and several process parameters are adapted in order to optimize and stabilize IBN production.

IBN is gaseous at the operating temperatures, and exits in the fermentation off-gas, which is sent to the purification unit. There, the off-gas is first washed from the fermentation material that could potentially be carried over. The IBN is then adsorbed in a solvent in a first column and, after that, desorbed in a second column, condensed and stored. Condensation occurs at a temperature below the boiling point of IBN, but high enough to avoid the condensation of other remaining gases. When needed, the liquefied IBN is recovered in pressurized TPED cylinders allowed for shipping.

2.4. Production at Demo scale

Three WSH-based runs were realized at the Demo scale for IBN production, and several technical issues were encountered

- Clogging of substrate flowmeter (leading to WSH feeding arrest) was observed for the first two runs, liked with the handling of the WSH substrate. Such clogging, as well as fouling, has been observed previously at the Pilot scale (500L, Pomacle) but could then be alleviated.
- Contaminations were observed, rather late for the first run and earlier for the second one, and we believe that our standard cleaning was not adequate for the clogged lines and should be improved for such feedstock.

These issues limited to 67kg the production of WSH-based IBN during this first campaign. Based on studies at the lab scale, mild pasteurization (use of temperatures lower than 120°C) appears today as the best potential way to solve this important issue. Altogether, the use of better strains produced in between and validated at the lab scale, and of a milder pasteurization process for WSH substrate preparation should significantly improve production in the next campaigns.

2.5. Analysis of 2G IBN

Analysis have been realized on the WSH-based IBN, IBN content after normalization was measured at 99,91% and the detected impurities are listed in Table 2. Sulphur compounds and oxygenates were identified as impurities that need to be trapped before PIBs' or Oligomers' production Furthermore, some solvent coming from the purification process was found in the bio-IBN: this does not deter from converting the bio-IBN into PIBs or Oligomers but could affect the quality of end-products. These issues delayed the delivery of "100 kg scale" to Ineos for test in their pilot plant.

Compound	Comment
Purification process solvent	Currently being fixed

Sulphur compounds	DMD, DMDS
Oxygenates	acetone, tert-Butanol, ethyl-methyl-ketone, methanol
Water	
Hydrocarbons	

Table 2. impurities identified in the bio- IBN from the Leuna Demo-plant.

3. Conclusions

After successful trials at the pilot scale, A first series of trials was conducted in Leuna at the demo scale with WSH as substrate, but several technical issues had to be overcome, which limited IBN production, to 67 kg of IBN during this campaign. Production was sufficient to identify impurities in the bio-IBN, including compounds which could impact IBN conversion into di- or poly-isobutene. Thus, downstream processing of IBN should be refined, in order to remove them.

In order to minimize the clogging and fouling issues while preserving sterility, mild pasteurizations, already validated at the lab scale appears as the most promising solution. One should also note that since these first trials in 2018, new IBN production strains with considerably better performances are available (not shown in this report), and should significantly contribute to the achievement of higher productions.

The presence of various impurities potentially capable of impacting the conversion of IBN is another aspect to address. As of today, partners GBE and INEOS have agreed to postpone the first trials of conversion of 2G IBN into DIB or PIB at the Pilot scale. Several treatments are envisioned to get rid of the impurities, and the pilot-scale conversion of IBN in DIB and PIB will be resumed as soon as the best solution is identified and applied.