More transport fuels from organic residues

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Preface

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Photo cover: Petrol station, from Shutterstock

Abbreviations

AB	Acetone and butanol
ABE	Acetone, butanol, ethanol
IBE	Isopropanol butanol and ethanol
GHG	Greenhouse gas
HDO	Hydrodoxygenation
PS	Paper sludge
PSA	Pressure swing adsorption
SAF	Sustainable Aviation Fuel
TPV	Thermo-pervaporation
SSF	Simultaneous saccharification and fermentation

1 Summary, approaches and goals

The project “More Transport Fuels from Organic Residues” (MOTOR) aims to produce advanced biofuels for the transport sectors such as aviation, shipping and long haul, where electrification or gaseous biofuels will not generate the reduction of their carbon footprint in the short term. Wet lignocellulosic waste streams that do not compete with food/feed market are used as the feedstock for the production of the advanced biofuel. The approach followed in the project is shown in Figure 1.

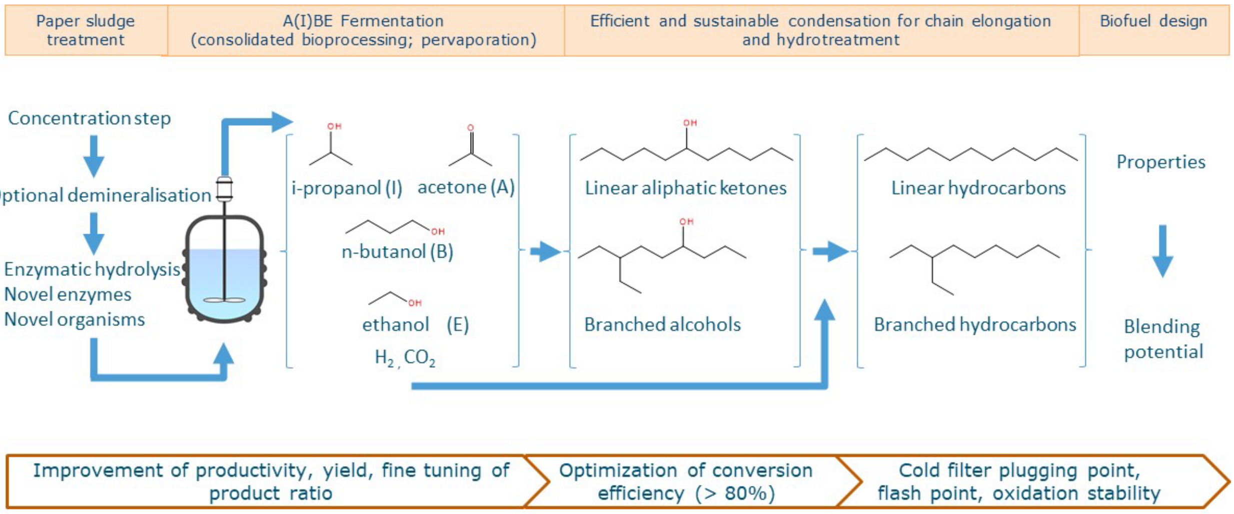

***Figure 1** Valorisation of paper sludge and 2nd generation biomass to biofuels in MOTOR.*

The MOTOR project is a collaboration between partners that cover the entire value chain from paper sludge source (Smurfit Kappa Parenco BV), paper sludge treatment (TNO), paper sludge enzymatic hydrolysis (IMEnz Bioengineering BV) and fermentation (Wageningen Food and Biobased Research, WFBR), alcohol conversion into intermediates (WFBR), hydrogen purification (HyGear Technology and Services BV), and hydrogenation to final products (Kuwait Petroleum Research and Technology BV, KPRT). Assessment of the integrated system was done by covering techno-economics, sustainability, market assessment and fuel properties (TNO and Goodfuels).

In MOTOR, innovations are combined in the fields of:

1. Substrate hydrolysis and fermentation combined in one step. Use of paper sludge as a source for production of IBE (a mixture of isopropanol, butanol, ethanol) with modified bacteria to enable direct utilisation of cellulose by consolidated bioprocessing (no need of external enzymes)
2. Smart downstream processing of the fermentation products is performed by thermo-pervaporation and extraction
3. A heterogeneous catalytic process is applied for formation of long chain aliphatic ketones from the alcohol mixtures
4. The aliphatic ketones are hydrotreated into iso-paraffins. The internal use of hydrogen produced during the fermentation step has been evaluated.

As a feedstock, paper sludge from a recycling paper facility was used, as a model for other cellulose-containing streams. Paper sludge represents a side stream from paper mills or paper recycling were limited valorisation routes are currently implemented, and that is widely available. The paper sludge sample contained approx. 20% carbohydrates, mostly as the polymer cellulose. Sugars were released by enzymatic hydrolysis from the polymers in the paper sludge using available and novel enzymes and microorganisms. Subsequently these sugars were converted to a mix of isopropanol, butanol and ethanol (IBE) by fermentation using *Clostridium* strains. Both the hydrolysis and the fermentation processes were hampered by inhibitors in the paper sludge stream, mostly inorganic components derived from ink.

Various treatments were applied to the paper sludge to evaluate whether these components could be selectively removed. However, only a physical treatment, wet sieving, resulted in a better hydrolysable/fermentable substrate.

Downstream processing of IBE was shown in a new hybrid concept of thermo-pervaporation (TPV) of a model solution of IBE in water and subsequent extraction of the concentrated IBE solution with heptane. From this IBE mixture in heptane, mostly n-alkanes, i.e. linear C-11 and branched C-8, were produced by chemo catalytic conversion and subsequent hydrodeoxygenation (HDO) of the intermediate long chain aliphatic ketones. Hydrogen is produced in the fermentation and chemo catalytic conversion and is purified by PSA to be used for the hydrodeoxygenation step.

Both preliminary techno-economic and life cycle assessments (LCA) have been carried out, based on experimental data obtained from the main conversion steps in the process. The data were combined in a successful conceptual process design for a full-scale commercial facility. The calculated GHG emissions of SAF produced through the MOTOR process are estimated to be significantly lower than those associated with fossil-based jet fuel, making the MOTOR biofuel to have potential to be further developed as an advanced biofuel. In addition, the potential market demand and the perspectives for implementation of the MOTOR process have been evaluated (section 2.4). The technology readiness levels (TRL) of the technologies for advanced fermentation and chemo-catalytic condensation of alcohols are estimated at 3, based on the results obtained in this project and on literature reports and more research, mostly towards specific applications and adaptation of technologies, is needed to increase the commercialisation potential of this value chain for advanced biofuels.

2 Results and perspectives for application

2.1 Paper sludge as substrate for fermentation

The paper sludge samples used in the project were obtained from a paper recycling factory, and therefore, in addition to the standard composition of paper sludge, contain impurities derived from ink. A typical composition of paper sludge used in this project is shown in Table 1.

Table 1 Typical composition of paper sludge used in this study.

Component	% of dry matter
Glucan (mostly as cellulose)	16.4
Xylan	2.5
Other sugars	2.4
Total lignin	16.7
Ash	55.5

The high content in ash reflects the high content of inorganics in the streams, likely derived from chemical additives such as inks and fillers. The high content in ash reflects the high content of salts in the streams. These can be inhibitory to the action of enzymes to solubilize the sugars in the sugar polymers in the biomass, or to the microorganisms that ferment these sugars. Various methods for ash removal were evaluated experimentally. The most successful method was to wash the paper sludge was washed by wet sieving, resulting in samples that were more accessible for enzymatic hydrolysis of the sugar polymers, and with better fermentability. In Figure 2, the material before and after wet sieving is shown. The wet sieving treatment caused a significant loss of sugars in the samples, and therefore this method needs to be further optimised, or a different method could be used to remove salts.

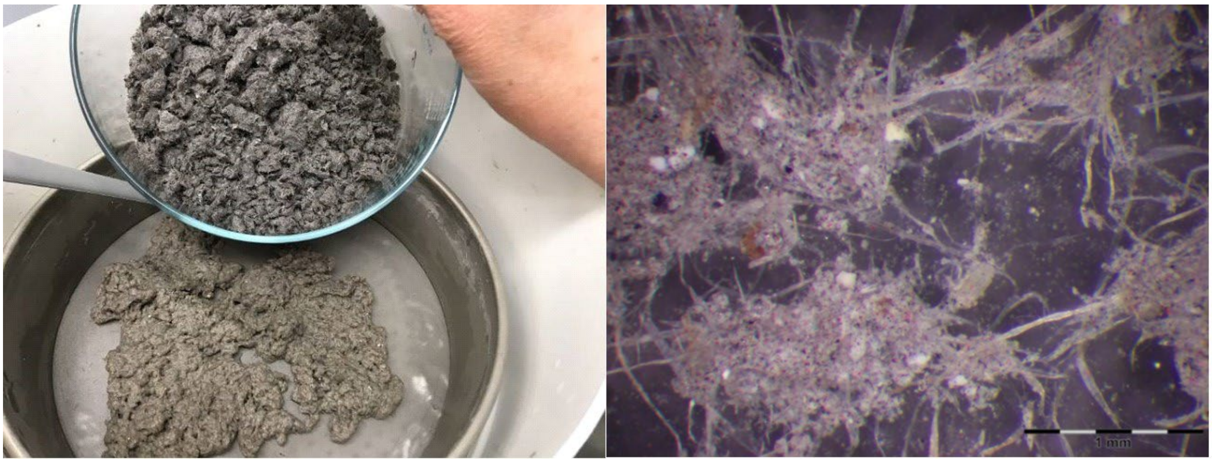

Figure 2 (left) Dry sludge before (above) and after (below) wet sieving. (right) Microscopy image from dry sludge before wet sieving.

Enzymatic hydrolysis of the paper sludge samples was screened using the IMENZ Bioengineering BV enzyme collection. This collection includes commercial enzymes and enzymes produced by cellulolytic microbial strains isolated from a range of sources. Some of the enzymes tested showed better glucose solubilisation, and these were used to produce hydrolysates to be tested for fermentation. In Figure 3, an example of results of paper sludge degradation by enzyme samples is shown.

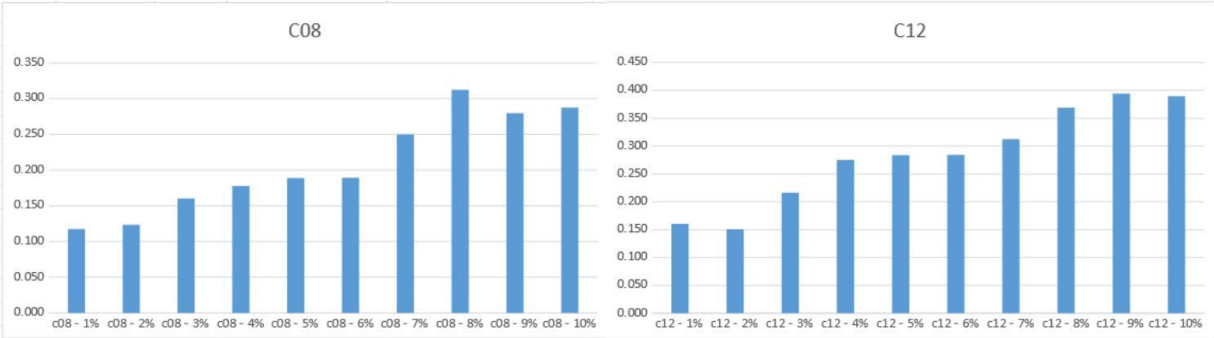

Figure 3 ***Glucose release from paper sludge (PS) as a function of enzyme concentration.. PS (20 % of dry matter) was incubated with increasing amounts of enzymes C.08 or C.12 for 2 h at 50 °C. The released glucose is given in an arbitrary unit.***

The conversion of sugars in paper sludge (PS) hydrolysate to a mix of isopropanol, butanol and ethanol (IBE) was carried out by *Clostridium beijerinckii*, an anaerobic bacterium that is well known for its capacity to convert sugars into isopropanol, butanol and ethanol (IBE). The soluble sugars in the PS-medium were converted into IBE (Figure 4). Both monomeric glucose and xylose were consumed and an IBE mix was produced at yield higher than 0.3 g IBE/g sugar consumed, indicating that other sugars or oligosaccharides in the hydrolysate were consumed as well.

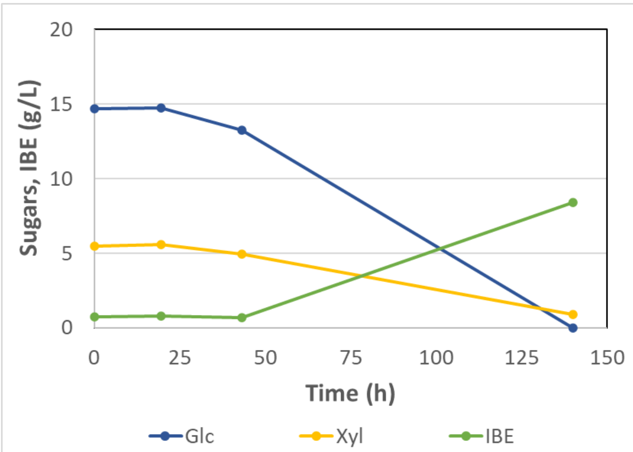

Figure 4 ***Production of isopropanol, butanol and ethanol (IBE) by C. beijerinckii on PS-hydrolysate supplemented with glucose (10 g/L). Abbreviations: Glc, glucose; Xyl, xylose; PS, paper sludge.***

The fermentation of paper sludge has been tested in different configurations. For tests of simultaneous saccharification and fermentation (SSF), paper sludge was incubated with commercial enzymes and the fermenting microorganism simultaneously. In these experiments acetone and butanol (AB) were produced by *C. beijerinckii* to a concentration of 4.7 to 5.1 grams total AB per litre. The time of inoculation, either at the start of the incubation of paper sludge with enzymes or 5.5 h thereafter did not result in a significant difference in AB production. Besides AB butyric acid was produced as a main product. From day 2 to 7 of the SSF experiment gas production was observed especially in samples inoculated with *C. beijerinckii* showing that hydrolysis and fermentation of paper sludge biomass continued in this period (Figure 5).

Figure 5 Simultaneous saccharification and fermentation of washed paper sludge in mixtures with commercial cellulases and C. beijerinckii (flasks 2 and 3). Flasks 1A and b correspond to the negative control, not inoculated. Vigorous gas production is observed in flasks 2 and 3, indicating growth of C. beijerinckii.

2.2 Production of fuels from fermentation products and process design

The approach followed in the MOTOR process, including the production of fuels from fermentation products is shown below (Figure 6).

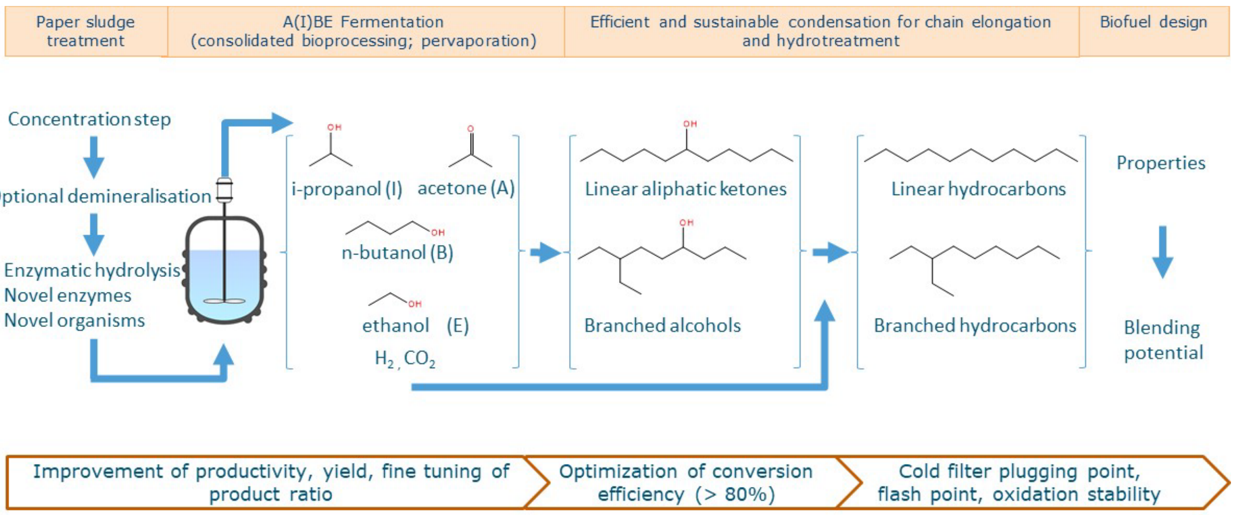

Figure 6 Approaches in MOTOR for valorisation of paper sludge to biofuels.

Long chain aliphatic ketones were produced from a synthetic mixture of IBE in heptane using heterogeneous catalysts. Various catalyst/support combinations were screened for optimal conversion and product distribution characteristics. C7-to-C15 components were obtained with selectivity towards C11-C15 chain lengths at a temperature of 145°C and autogenous pressure. A typical product distribution is depicted in Figure 7.

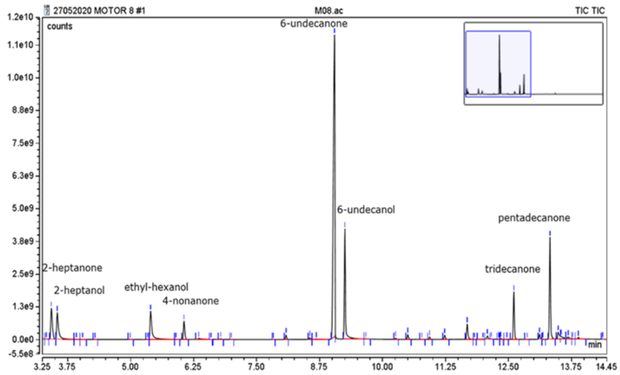

Figure 7 Product distribution of IBE conversion to aliphatic ketones.

The aliphatic ketones and alcohols are subsequently hydrogenated. Full conversion (>95%) of the ketones and alcohols in a synthetic mixture was demonstrated using a supported noble metal catalyst. The amounts of by-products formed was not significant and no significant formation of coke was observed.

The process flow diagram developed is depicted in Figure 8. The feedstock used for the MOTOR process is a paper sludge waste stream from a paper recycling mill, but the concept could be applied to other cellulose-containing streams as well, including those from 2nd generation biorefineries, depending on their specific composition.

The paper sludge feed stream can optionally undergo demineralisation/detoxification. In the base scenario this was not implemented. The resulting stream is sterilized (not depicted) and fed to a (fed) batch fermenter. In the bioreactor, the resulting stream fermented by *C. bejierinckii* following aconsolidated bioprocessing approach (CBP). In particular, the cellulose in the paper sludge is hydrolysed by enzymes produced by the microorganisms, and the resulting sugars are fermented to IBE. The fermentation product is filtrated to remove the biomass. The diluted aqueous IBE stream is first fed to a membrane pervaporation step to produce a concentrated aqueous IBE stream and an organic IBE stream. The permeate stream contains approximately 50 w% water from which the IBE is further separated by extraction with hexane that also serves as the solvent required by the downstream process.

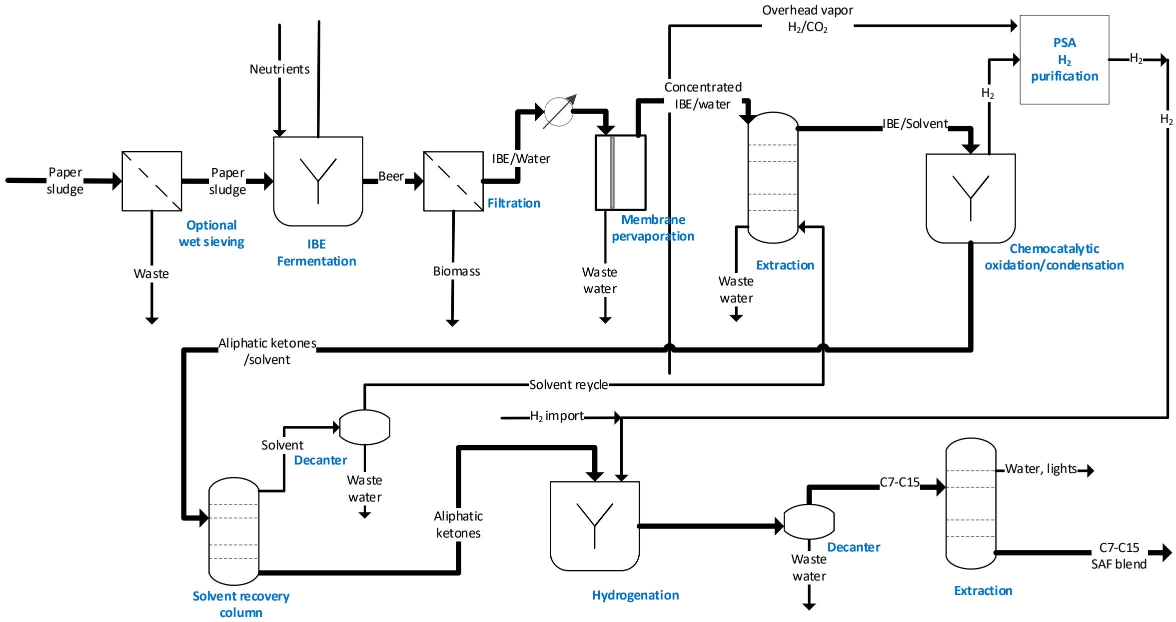

Figure 8 Process flow diagram of the MOTOR process.

The IBE is converted into linear aliphatic ketones (C7-C15) using a heterogeneous catalyst. The solvent is recovered from this stream by distillation and is recycled. The aliphatic ketone stream is then hydrotreated in the presence of a heterogeneous catalyst to obtain linear and branched alkanes. The hydrotreatment step utilises H_2 produced in the fermentation as well as in the dehydrogenation step.

Both streams contain small amount of organics and especially the stream from fermentation contains a large amount of CO_2. Pressure swing adsorption (PSA) was selected to purify the H_2 up to the hydrogenation feed specifications. Finally, the linear alkane product stream is purified in by distillation. The product obtained, based on the predicted composition, would have potential application as a sustainable aviation fuel (SAF) stream suitable for blending to obtain the required SAF specifications.

The process was modelled in the flow sheeting tool Aspen Plus, where experimental data were translated into full industrial scale, fully developed technology. Results show that the SAF product stream consists of C7-C15 stream predominantly consisting of linear C11 and branched C8 alkanes. The share in of C11 product could potentially be increased by increasing the amount of isopropanol obtained from fermentation. The mass yield in cellulose to SAF is 13%. Losses were found to be highest in the fermentation, filtration and membrane separation steps. These losses were due fundamental limitations in the reaction stoichiometry and in the conservative estimation of the conversion and fundamental limitations in membrane separation. The losses in extraction and conversion into alkanes were found to be relatively low.

Based on the technologies and data collected during the project, an LCA has been performed to determine the potential reduction of GHG emissions. The calculated GHG emissions of SAF produced through the MOTOR process are estimated to be significantly lower than those associated with fossil-based jet fuel (Figure 9), making the MOTOR biofuel to have potential to be further developed as a an advanced biofuel.

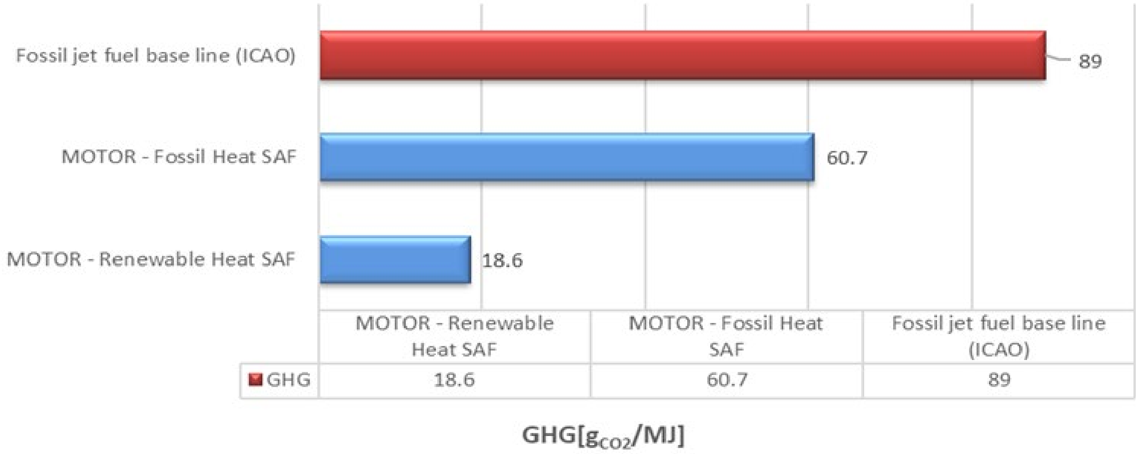

Figure 9 ***Estimated GHG emissions of the MOTOR process compared to the current fossil fuel-based jet fuel.***

2.3 General conclusions

The results obtained during the MOTOR project show the potential for a new route for SAF production, based on waste paper sludge, an organic wet residual stream that is widely available. The biofuel produced in the MOTOR project has properties that could potential as a SAF, however the catalytic routes developed need to be further optimised. From the results of the project, several conclusions can be made:

- Paper sludge streams where fermentable by the strain tested for IBE production, opening possibilities for further developments. The paper sludge sample used in this project is recalcitrant feedstock for saccharification using enzymes. Inorganic and other components need to be removed before saccharification and fermentation. To achieve a consolidated bioprocessing fermentation process further research to improve hydrolysis and fermentation titers is needed. During the project, interesting microorganisms and enzymes have been identified with increased paper sludge hydrolysis properties.

- Chemical catalysis of the fermentation products yields aliphatic compounds in range of C7-C15, with predominantly C8 and C11, in line with uses as fuel.

- Near full conversion was obtained for the aliphatic ketones and alcohols produced using a supported noble metal catalyst.

- A conceptual process design was made for the MOTOR process, and this was used for calculating mass and heat balances. Further improvement of the chemo-catalytic conversion step as well as tailoring the ratio of isopropanol/butanol/ethanol in the IBE stream are important for further optimisation.

- The first estimations of the GHG emissions associated with the MOTOR process show that a significant reduction could be possible as compared to fossil based fuels using renewable heat sources.

2.4 Perspectives for implementation: challenges and next steps

During the project, a market assessment has been conducted to identify potential challenges that MOTOR's sustainable aviation fuel will face during market uptake. In the MOTOR process, waste paper sludge is used as a model feedstock for production of jet fuel via an alcohol-to-jet- type process. Waste paper sludge was selected for its high cellulose content and because it is considered a second-generation feedstock, which does not compete with the production of food or feed. Therefore, it can potentially be used for production of advanced biofuels. The market assessment brings insights to the process of technology optimisation market, making the proposed fuel a market alternative.

Based on the market assessment using sustainable aviation fuel (SAF) as main product of the MOTOR process, with paper sludge as feedstock, several challenges regarding implementation have been identified:

Integration of the MOTOR concept within the existing paper mill infrastructure
The MOTOR process is a promising strategy to promote circular economy in the pulp and paper industry while adding value to streams once considered as waste. The future of paper sludge as potential feedstock depends on the efficiency with which the pulping process evolves towards higher fibre recovery, yet the residual stream will hardly disappear. Possible changes in the process and how those may affect MOTOR must be mapped while working on a business plan. Moreover, it is essential to assess the impacts of centralized and decentralized production in terms of OPEX and CAPEX to better design the process.

Integration of MOTOR products within the fuel pool
In MOTOR mostly linear and saturated carbon chains are being produced. Preliminary results indicate that MOTOR based SAF is predominately composed of n-alkanes, i.e., 45 wt% C-11 and 51 wt% C-8. The high content of C8 n-alkanes might lead to an overall off-spec cetane number.

Assuming that the fuel composition will remain as such, the most effective way to integrate it within the fuel pool is exploring it as fuel blending component rather than its neat use. Nevertheless, concrete assessment on the fuel specifications should be further addressed following the properties provided by ASTM. *Next steps required in the development and scale-up of this technology*

In order to bring this process to the market it is necessary to consider the following steps:
1. Identify and solve bottlenecks within the process for cost reduction and optimisation
2. Define/ guarantee feedstock supply
3. Form a consortium involving potential stakeholders to support the certification process and requirements
4. Start testing of fuel against ASTM specifications, using a surrogate solution while scale of biomass-derived fuel is not sufficient. Redesign process tuning product composition to meet specification
5. Asses blending potential
6. Technical due diligence for scale up production
7. Design best fit with the paper mill industry and supply-chain to minimize cost and emissions while securing feedstock availability.

Key actors in the exploitation and implementation of the concept and incentives and steps to involve them in further development and commercialisation
- Paper mill industry: product and segment diversification adding value to core business. They should be involved in the initial stages of R&D given the feedstock dependency and potential benefit. Retrofitting MOTOR's process to a current mill would also benefit from lower CAPEX
- OEMs: increasing the pipeline of new fuels and green marketing. Valuable partners to advise during/pre certification
- Engineering partners: knowhow building, useful for other projects on renewable bioenergy. Involved during scale-up process as project partner or service provider
- Commercial partner, fuel providers, aviation companies, airports (assess/advise/facilitate on market implementation strategy and uptake): direct benefit from low carbon fuel supply. Commercial partners should be involved from R&D phase. Frontrunner position is a valuable gain during the energy transition.

In summary, SAF is considered an essential strategy in the short and medium term for the aviation sector to meet their climate targets. However, the market is very competitive, affected by the small number of players and by the high prices of SAF compared to conventional petrochemical jet fuel.

In order to allow MOTOR's fuel to reach the market the following points need to be achieved:
- Meet technical requirements: currently MOTOR's fuel is still in development, but it is necessary to address the specifications provided on Tier 1 and 2 tests during ASTM certification while designing the process
- Scale up: the security of the supply is vital for a fuel uptake. Lack of supply capacity is one of the main drawbacks of HEFA. MOTOR has the advantage of having a sustainable feedstock according to the RED II. Yet its supply its not guaranteed as the paper industry has other potential end uses for paper sludge. Uptake agreements are necessary. Otherwise, the use of alternative or mixed feedstocks, whose supply can be guaranteed, such as agricultural side streams, energy crops or side streams from food processing, needs to be explored.
- ASTM Certification. Once point a) and b) have been addressed, the ASTM certification is needed to guarantee access to the market
- Legislative support. SAF is not likely to achieve the cost-effectiveness of its fossil counterpart. Therefore, legislative and policy support will be needed to cope with the high costs
- Sustainability. Design the process to lower the GHG emissions as much as possible. Additionally, guarantee a traceable and sustainable value-chain for certifications purposes.

3 Contribution of the project to the goals of the program

Increasing the sustainability and reducing the GHG emissions in the transport sector is an important target in the Renewable Energy Directive II (REDII). In this frame, the International Air Transport Association (IATA) and the Air Transport Action Group (ATAG), together with organizations such as the International Civil Aviation Organization (ICAO), have committed to reduce their emissions by: a) improving fuel efficiency by 1.5% per year; b) from 2020 onwards achieve carbon neutrality; c) 50% emission reduction by 2050 (with respect to 2005 levels) [1 2]. Theses ambitious targets require new ways of dealing with energy requirements, which currently heavily depends on liquid fuels, and will remain the same for the foreseeable future. Some low-carbon options, such as electrification or hydrogen, are not likely to be implemented at commercial scale in the near term due to the current mismatch between the needs of the aviation sector and the status of technology development for such alternatives. Sustainable Aviation Fuels (SAF), are considered the best readily available technological option to reduce the environmental impact of aviation, in the short-term. SAF are defined as non-fossil derived aviation fuels, i.e. a sustainable alternative to jet fuels, and need to be drop-in, i.e. chemical and physical properties similar to those of conventional jet fuel. Consequently, SAFs can be mixed with and use the same infrastructure of fossil-based jet fuel[3].

Among SAF, biofuels are spotted in favourable position to achieve long-term emission reduction in the aviation sector[2]. Synthetic jet fuels are also considered as SAF, yet their production is still limited and costly. Thus, SAF fuels are the most cost-effective scale up alternative in the short term[1]. Biofuels have a huge potential to reduce GHG emissions, on a well-to-wheel basis, compared to fossil jet fuels. Yet this potential may vary depending on the feedstock and the technological conversion pathway implemented. Furthermore, it is also vital that production and consumption of bio jet fuel increase in order to achieve the GHG emission-reduction targets. The current SAF production quantities are too low to meet the mentioned goals, and therefor there is a need for alternative routes, complying with the new sustainability requirements.

The results obtained during the MOTOR project show the potential for a new route for SAF production, based on waste paper sludge, an organic wet residual stream that is widely available. The biofuel produced in the MOTOR project has properties that could potentially generate a SAF, however the catalytic routes developed need to be further optimised.

The lifecycle analysis (LCA) of this route shows a reduction in GHG of using sustainable heat sources and further developing different steps in the process, the process could achieve a potential reduction of more than 75% compared to conventional kerosene production. This reduction is sufficient to satisfy the sustainability target in the REDII directive. The use of renewable heat sources here is essential, since it the reduction is only 50%

The MOTOR process has been developed in collaboration between Research Institutions (WFBR, TNO) and small (IMENZ Bioengineering bv, HyGear Technology and Services bv, GoodFuels bv) and big industries (KPRT, Smurfit Kappa). A first proof of principle has been delivered during the project implementation.

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Ibrahim, A. B. A., & Akilli, H. (2019). Supercritical water gasification of wastewater sludge for hydrogen production. International Journal of Hydrogen Energy, 44(21), 10328–10349. https://doi.org/10.1016/J.IJHYDENE.2019.02.184

4 Public publications from the project

Oral presentation by Maarten van Haute, Kuwait Petroleum Research and Technology
Circulair Congres, 8 May 2019
MOTOR More transport from organic residues
https://www.biobasedeconomy.nl/wp-content/uploads/2019/05/MOTOR.pdf

Oral presentation by Maarten van Haute, Q8 Kuwait Petroleum Research and Technology
"The urgency and requirements for renewable building blocks from waste streams"
Events by Institute for Sustainable Process Technology ISPT, Maart 15, 2022, Soest
https://ispt.eu/events/problem-solving-and-matchmaking-waste-stream-valorisation/

Oral presentation by Pieternel Claassen, WFBR
Microbial conversion of biomass, The integrated approach at WFBR
P. Claassen, T. de Vrije, A. Lopez-Contreras, C. Boeriu, M. Werten, J. van Medevoort, N. Kuipers, R. Pazhavelikkakath Purushothaman
BIT's 3rd Industrial Biotechnology Congress, October 25, 2019 Singapore

Oral presentation by Pieternel Claassen
MOTOR: Integrated bioprocess for conversion of biomass
P. Claassen, T. de Vrije, A. Lopez-Contreras, C. Boeriu, D. van Seijst, M. Werten, J. van Medevoort, R. Pazhavelikkakath Purushothaman
IEA Biohydrogen Symposium, December 19, 2020 Taichung

Oral presentation by Ana López Contreras, WFBR
"Process development for production of biofuels from paper sludge via fermentation and chemo-catalytic conversions" by A. M. López-Contreras, J. W. Dijkstra, T. de Vrije, K. Dussan, S. Luzzi, B. van den Burg, M. Budde, D. van Seijst, J. van Medevoort, R. K. Pazhavelikkakath Purushothaman, J. van Haveren, O. M. Morales-González, F. Ferrari, M. Rep, A. Hakeem, M. Van Haute and P. A. M. Claassen
EUBCE Congress, May 11, 2022, Online
Proceedings publication at EUBCE 2022, available at http://www.etaflorence.it/proceedings/ (after free registration)

Oral and poster presentation by Ana López Contreras, WFBR
WUR Knowledge Base Programme "Circular and climate neutral society"
KB34 011 005 MOTOR: More transport fuels from organic residues
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5 Project partners

To explore the potential of nature to improve the quality of life

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