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# BioMates

Deliverable D2.5 - Report on product stabilisation and storage guidelines

*Version 01*

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## Summary

The objective of this document is to report stabilization and storage guidelines of *bio*-based inter*mediates produced* by ablative fast pyrolysis (AFP) and mild catalytic hydroprocessing (mild-HDT) enabling the technology upgrade to a pre-commercial level with respect to 1) energy integration, 2) feedstock variability management, and 3) product stability during storage. The BioMates product employed in this study was the hydrotreated bio-oil from straight-run ablative fast pyrolysis of straw provided by UCTP.

This document contains the results of the extended 12-month storage study and the accelerated ageing experiments of the BioMates products enabling the assessment of their stability and the recommendation of their storage guidelines. Feedstock variability has been assessed by analysing bio-oils of different origin obtained by ablative fast pyrolysis of the biomass feedstocks.

The document also outlines important aspects to be considered with respect to energy integration, an area driving the overall efficiency. As hydrotreating processes are exothermal, the surplus energy from the hydrotreating can favourably get used to increase temperature of feed streams. One aim of project BioMates is the identification of an optimum between lowest net energy consumption and lowest operating cost. Commercial success depends on low operating costs, so not each and every energy integration proposal for reduced energy consumption might be commercially justified.

## Contents

# 1. Introducing BioMates

## 1.1. The BioMates Project

The BioMates project aspires in combining innovative 2nd generation biomass conversion technologies for the cost-effective production of *bio*-based inter*mediates* (BioMates) that can be further upgraded in existing oil refineries as renewable and reliable co-feedstock. The resulting approach will allow minimisation of fossil energy requirements and therefore operating expense, minimization of capital expense as it will partially rely on underlying refinery conversion capacity, and increased bio-content of final transportation fuels.

The BioMates approach encompasses innovative non-food/non-feed biomass conversion technologies, including **ablative fast pyrolysis (AFP)** and single-stage **mild catalytic hydroprocessing (mild-HDT)** as main processes. Fast pyrolysis in-line-catalysis and fine-tuning of BioMates-properties are additional innovative steps that improve the conversion efficiency and cost of BioMates technology, as well as its quality, reliability and competitiveness. Incorporating **electrochemical $H_2$-compression** and the state-of-the-art **renewable $H_2$-production** technology as well as **optimal energy integration** completes the sustainable technical approach leading to improved sustainability and decreased fossil energy dependency. The overall BioMates-Concept is illustrated in Figure 1.

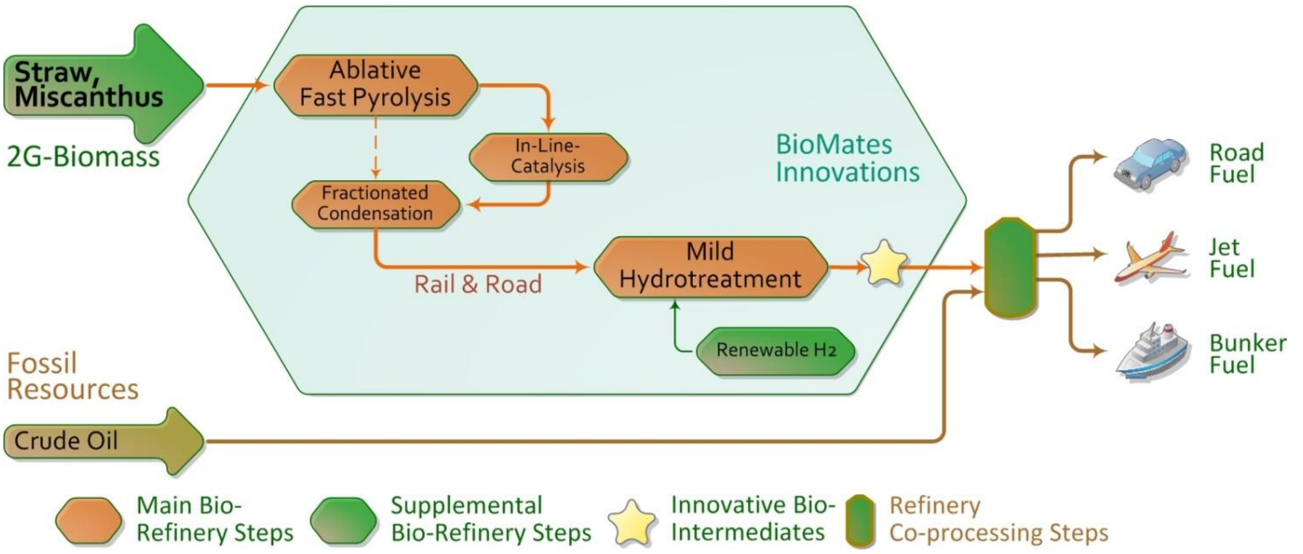


Figure 1: The BioMates-concept

The proposed technology aims to effectively convert residues and non-food/feed plants or commonly referred to as 2nd Generation (straw and short rotating coppice like miscanthus) biomass into high-quality bio-based intermediates (BioMates), of compatible characteristics with conventional refinery conversion units, allowing their direct and risk-free integration to any refinery towards the production of hybrid fuels.

## 1.2. European Commission support

The current framework strategy for a Resilient Energy European Union demands energy security and solidarity, a decarbonized economy and a fully-integrated and competitive pan-European energy market, intending to meet the ambitious 2020 and 2030 energy and climate targets /EC-2014a, EC-2014b/. Towards this goal, the European Commission is supporting the BioMates project for validating the proposed innovative technological pathway, in line with the objectives of the LCE-08-2016-2017 call /EC-2015/. This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 727463.

### 1.3. The BioMates team

The BioMates team comprises eight partners from industry, academia and research centres:

- Fraunhofer Institute for Environmental, Safety, and Energy Technology UMSICHT, Germany (Project Coordination) - *www.umsicht.fraunhofer.de*
- Centre for Research & Technology Hellas / CERTH - Chemical Process & Energy Resources Institute / CPERI, Greece - *http://www.cperi.certh.gr/*
- University of Chemistry and Technology Prague, Czech Republic - *http://www.vscht.cz*
- Imperial College London, United Kingdom
  *www.imperial.ac.uk*
- Institut für Energie und Umweltforschung Heidelberg GmbH / ifeu, Germany - *www.ifeu.de*
- HyET Hydrogen B.V. / HyET, The Netherlands - *www.hyethydrogen.com*
- RANIDO, s.r.o., Czech Republic
  *http://www.ranido.cz/*
- BP Europa SE, Germany
  www.bp.com/en/bp-europa-se.html

For additional information and contact details, please visit *www.biomates.eu*.

## 2. Preface

This document encompasses the assessment of the activities related to the Task 2.6 objectives of WP2 of the BioMates project. More specifically, the BioMates products stabilization and storage guidelines are investigated addressing also the substantial commercial challenges referring to the energy integration schemes in a conventional refinery and the feedstock variability impact on the BioMates products quality. This deliverable was carried out by BioMates working group, led by BP and supported by UCTP and CERTH. Section 4 describes the potential energy consumption improvement via applying optimal energy integration in the refineries, while section 5 describes the impact of the variable feedstock quality on the BioMates product quality. Moreover, the BioMates products stability study is presented thoroughly in section 6, analysing both the extended storage study and the accelerated ageing experiments, as well. Section 7 summarizes the conclusions of the aforementioned tasks of this deliverable.

## 3. Deliverable verification

The verification of D2.*5 Report on product stabilization and storage guidelines* is based on the production of BioMates product (hydrotreated bio-oil from straw) at UCTP (~3.5 litres) that was delivered to CERTH (on 15/12/2017) for the extended 12-month storage study. In addition, for the requirements of the accelerated ageing experiments of the BioMates products, upgraded products of bio-oil (from straw) via catalytic hydrotreatment produced at CERTH were employed.

## 4. Energy integration schemes in conventional refineries

### 4.1. General aspects

Any ambition to enhance respectively reduce energy consumption by energy integration of a conventional refinery needs to address the most important aspect first: a status report of the individual energy consumption of each process unit, detailed mass flow data, and the specific properties of the streams going in and out of each process unit to allow proper assessment.

The amount of energy used per volume of crude processed in conventional refineries is one of the key differentiators. Since decades refineries have established a multitude of ways to balance out the energy demand of cold process streams requiring heating up with hot process streams in need of cooling. Therefore one of the first areas to tackle is heat management systems, which by improved design of assets and pipework help to optimize energy flows. An example is tailored steam systems for a systematic use of surplus heat, potentially in combination with reduced pressure of the steam systems to reduce losses. Or substitution of passive steam pressure regulators with micro-turbines, which reduce the steam pressure accordingly, but as a side effect generate electricity for own consumption or the local electric grid.

Many product streams require a certain temperature level for best efficiency of the process units. A crude distillation unit (CDU) for example requires the crude to be heated up sufficiently to allow proper functionality of the CDU. Mostly any CDU today use the excess heat of the products leaving the CDU to transfer thermal energy to the cold crude, which is finally warmed up in large furnaces to the specified temperature for the CDU entry point. The efficiency of those furnaces can be increased by better control of burners as well as optimized oxygen flow, better oven design, and improved maintenance schedule. Some typical refinery process units might benefit from newly developed catalysts, as their new chemistry might allow running processes at lower temperatures and/or lower pressure leading to improved efficiency.

Modern refinery process units are being designed and built differently than decades ago. Even modern systems use a gas torch to get rid of excess gas safely in cases outside of the standard window of operations, but in routine operations the reduced use of the torch is commercially and environmentally beneficial. With those gas recovery systems, less gas gets directly converted into $CO_2$, and more liquid product is generated.

As large amounts of liquids need to get pumped, any improved efficiency of electric pumps and drives helps to reduce the electric energy demand for those systems. During larger turn-arounds, often new pumps and mixers with lower rotational speeds, but with the same nominal throughput get installed, and those upgraded units demonstrate the potential of new hydraulic designs.

## 4.2. Energy integration of a mild-HDT assets within vicinity of an existing refinery

The core process for conversion of raw pyrolysis oils into a valuable intermediate is hydrotreating (HDT), an exothermic process requiring substantial amounts of hydrogen. Refineries processing fossil crude oils generate up to 100% of their specific hydrogen demand for hydrogen-consuming processes in certain assets such as naphtha reformers. Any additional hydrogen typically is being generated in steam reformers fed with steam and natural gas or refinery gases like propane & butane. The hydrogen gets used in different assets, and hydrogen contained in typical process off-gas is being purified and recycled. For mild-HDT, the hydrogen supply from the refinery would help to compensate $H_2$-consumption in the HDT and reactor quenching. Energy integration of a stand-alone mild-HDT focuses on use of existing hydrogen production and recycling assets, and the integration of the exothermic energy from HDT into the range of refinery assets in need of thermal energy.

It is important to assess energy-efficient use of existing utilities; among those are compressed air, fresh water supply, waste-water treatment, fuel supply and steam streams. Fuel oil or certain gas streams from the refinery could serve as energy source for the furnace, preheating the feed entering the unit or the unit itself. The available steam from an existing refinery network could act as stripping steam. As the steam production in larger units is more efficient than in smaller stand-alone units, energy integration would potentially help to enhance efficiency. The target is overall-maximised efficiency, lowest greenhouse gas emissions, and all of these at minimised operating costs.

### 4.3. Energy integration within a stand-alone mild-HDT asset

Even in a stand-alone unit, the conversion process of raw pyrolysis oils into intermediates allows energy integration. The aspects presented in the general paragraph above applies, and the exothermic HDT process would benefit from heat exchangers exchanging the thermal energy contained in hot products leaving the HDT and cold feed streams entering the HDT. Another source of energy available for integration is the energy contained in the high-pressure gaseous product stream, as that energy can be used for the compression of cleaned gas past purging and prior to the reactor. The liquid product stream recycling back into the reactor is an additional source of energy that can be employed for heating the fresh feed via heat exchangers, allowing direct heat integration within the unit itself.

In any proposed energy integration case two aspects need to be considered. The first aspect is the technical feasibility, the other the commercial feasibility. The final outcome of such an assessment depends on the scale of the unit, the specific feedstocks getting processed and external drivers like local regulations, local energy prices, overall GHG assessment and others.

## 5. Impact of feedstock variability on the BioMates products quality

Bio-oil composition and its physico-chemical properties are key parameters both for its storage and for its further processing (by mild-HDT) and attainable final product quality. The properties and composition depend not only on the severity of biomass pyrolysis (operating conditions), but also on the kind and origin of biomass (straw /miscanthus) or bio-oil ageing. To address the feedstock variability issue, different bio-oils obtained by the AFP process (see WP1) were studied /BioMates-D1.4/ with the selected optimum catalyst(s) under the optimum reaction conditions to describe/determine the impact of the variable feedstock quality on the product quality to ensure/demonstrate that the optimum catalyst(s) and operating conditions are sufficiently robust. The variability of analysed feedstocks is demonstrated in the following **Fehler! Verweisquelle konnte nicht gefunden werden.**. It comprises data for bio-oils from different feedstock materials, generated within BioMates by ablative fast pyrolysis (AFP) in the straight-run mode (Deliverable 1.1) and with with staged condensation (Deliverable 1.2) /BioMates-D1.1/BioMates-D1.2/. Additionally, the properties of beech-wood-derived pyrolysis oil, generated outside BioMates, are presented for reference.

**Table 1:** Analytical results of bio-oils from different materials and pyrolysis operation modes, derived at UCTP /BioMates-D1.4/.

| Pyrolysis mode | | straight-run AFP | | AFP with staged condensation | straight-run AFP |
|---|---|---|---|---|---|
| Feedstock | | straw[a] | Miscanthus | straw[a] | Beech wood |
| Comment | | see D1.4, Table 3 | Alternative BioMates sample, results not reported yet | Sample from stage 1, see D1.4, Table 5 | BioMates-external material |
| Ultimate analysis | Unit | original samples | | | |
| C | wt.- %. | 52.36 | 51.106 | 55.07 | 43.3 |
| H | wt.- %. | 8.21 | 8.011 | 8.07 | 7.7 |
| S | wt.- %. | 0.06 | 0.017 | 0.05 | 0.1 |
| N | wt.- %. | 0.75 | 0.175 | 0.62 | 0 |
| O[b] | wt.- %. | 38.62 | 40.69 | 36.19 | 48.90 |
| Ultimate analysis | | pure organic content ("OC" - without water) | | | |
| C | wt.- %. | 67.44 | 64.92 | 68.58 | 56.97 |
| H | wt.- %. | 10.57 | 7.17 | 7.32 | 6.62 |
| S | wt.- %. | 0.08 | 0.02 | 0.06 | 0.13 |
| N | wt.- %. | 0.97 | 0.22 | 0.77 | 0.00 |
| O[b] | wt.- %. | 20.94 | 27.66 | 23.26 | 36.27 |
| Yield of OC | wt.- %. | 20.88 | 20.29 | 23.29 | 59.87 |
| Various parameters | | original samples | | | |
| Density at 15 °C | $g/cm^3$ | 1.107 | 1.1202 | 1.133 | 1.21 |
| Density at 20 °C | $g/cm^3$ | 1.1 | 1.1193 | 1.126 | |
| Density at 40 °C | $g/cm^3$ | 1.08 | 1.096 | 1.109 | 1.19 |
| Kin. viscosity at 20 °C | $mm^2/s$ | 884 | 903 | 640 | |
| Kin. viscosity at 40 °C | $mm^2/s$ | 140 | 215 | 106 | 87.6 |
| Water | wt.- %. | 22.36 | 21.28 | 19.70 | 24.5 |
| Micro Conradson carbonisation residue | wt.- %. | 15 | 19 | 15.4 | 21.8 |
| Carboxylic acid number - special method for bio-oils | mg KOH/g | 54.3 | 65.1 | 81.0 | 115.4 |
| Total carbonyls content | mmol/g | 3.08 | 4.09 | * | 3.88 |
| Total phenols content | mmol/g | 3.63 | 4.22 | * | 2.94 |
| Higher heating value | MJ/kg | 23.3 | 21.6 | 25** | 18.2 |
| Lower heating value | MJ/kg | 21.5 | 19.8 | | |
| Solids content | wt.- %. | 0.22 | 1.48/0.07 | * | * |

* not been measured yet  ** calculated

a Wheat / barley straw 50:50 wt.-%  b calculated to 100 %

It is obvious that there can be significant differences among the bio-oils due to both the different source of biomass used and different biomass treatment during the bio-oil production (i.e. single stage vs. multiple stage condensation of the pyrolysis vapours. From the practical point of view, the content of solids has been identified as critical for further bio-oil processing as it can cause fast plugging of the catalyst bed.

# 6. BioMates products stability assessment

The storage properties of the fuels are critical considering the introduction of new fuels into the markets and their successful commercialization, as well. With this in view, the stability of BioMates products during an extended 12-month storage period was studied in an attempt to define its storage guidelines. Accounting that the BioMates products are new competitive products originating from the co-processing of bio-based and fossil oil-based fractions, the issue of their storage stability has not been previously reported. However, the storage stability of bio-oil produced from biomass fast pyrolysis has been studied, affecting its further application and exploitation as a renewable transportation fuel and these findings will be briefly reported here. The bio-oil is composed of variant oxygenated compounds, and tends to age during storage leading to its quality deterioration impeding its subsequent refining and further application, as well /Chen-2014/. In particular, in long-term storage period the bio-oil components participate in various polymerization and polycondensation reactions creating oligomers with higher molecular weight. The storage stability of the bio-oil is commonly described via variations of the viscosity and the water content (WC) /Lu-2008/. Recently the research in the field of the bio-oil stability is being enriched towards improving its poor storage stability associated with viscosity reduction, elimination of the highly reactive compounds etc. /Lu-2008/Hilten-2010/ /Chen-2014/Yang-2015/. Furthermore, the ageing rate of the bio-oil under accelerated ageing conditions (exposed to high temperature) could be accessed in terms of the viscosity increasing rate /Lu-2008/.

In order to explore the BioMates products stability towards their commercialization this deliverable aims

- to provide specific guidelines for the extended storage of the BioMates products monitoring their quality variation, and
- to elucidate the ageing rates of the BioMates products under accelerated oxidation conditions (temperature, air effect, metal parts).

## 6.1. Extended storage study

For the purposes of the extended storage study the BioMates product employed was the hydrotreated bio-oil from straight-run ablative fast pyrolysis of straw, provided by UCTP[1]. In particular the BioMates product was delivered at CERTH and stored in Thessaloniki for 12 months at a plastic container outdoors under a shed at atmospheric temperature without exposure to sunlight. The storage study began on 15/12/2017 and ended on 15/12/2018. The weather conditions in Thessaloniki during the storage study were mild, having a rather soft winter and a not very hot summer (average annual temperature was ~16 °C). During the storage period, samples were taken on a monthly basis and the storage stability was studied with respect to total acid number (TAN, ASTM D 664) and WC (ASTM D 1744) according to a stability monitoring protocol developed previously by CERTH. Before the sampling the plastic container was shaken to ensure a homogeneous sample. The samples of the 12-month storage study are depicted in Figure 2.


Figure 2 : Samples of the BioMates products during the 12-month storage study

[1] Production and properties of straight-run bio-oil are described in the public deliverable D1.1/D04 "Straight-run AFP products from straw & Miscanthus" /BioMates-D1.1/

Figure 3 presents the monitored properties of the BioMates products during the 12-month study period. As it is depicted, the TAN values of the BioMates products during the storage study did not present noticeable variation, indicating that there is no increase in the acidity. This is in consistency with bio-oil ageing during storage as reported in the literature (Chen-2014). However, WC of the BioMates product followed a steadily increasing trend until the 5$^{th}$ month of the storage period and then significantly fluctuated until the end of the study (the analysis of the sample of the 2$^{nd}$ month was not conducted due to maintenance operations of the instrument). The WC increase during the storage study could be attributed potentially to reactions that generate water (e.g. esterification reactions results to formation of water) (Chen-2014). Therefore, it can presumably be inferred that the BioMates products storage is feasible for a maximum period of 5 months, whereas the regular monitor of their quality characteristics via WC analysis is recommended. Furthermore, we expect that the storage of BioMates products in colder climatic conditions than those typical for Thessaloniki in Greece would presumably not cause more severe degradation.

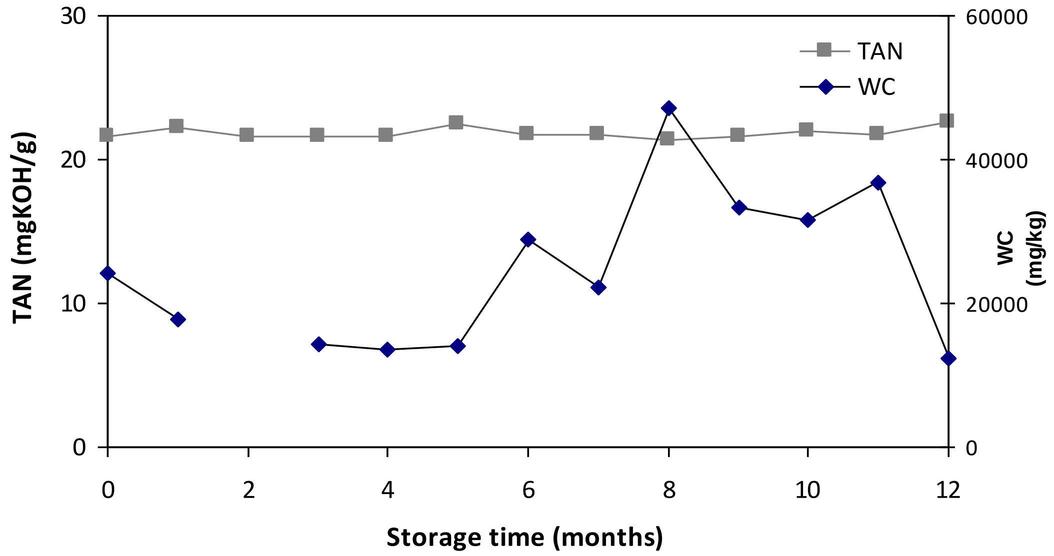


Figure 3 : WC and TAN of the BioMates product during the extended storage study

## 6.2. Accelerated ageing tests

The BioMates products stability was also assessed via accelerated ageing tests while observing the effects of ageing on their quality deterioration. These accelerated experiments of the BioMates products aim to further evaluate their storage limitations and parameters affecting it (temperature, contact with air and contact with metal parts). Furthermore, the accelerated ageing experiments of the BioMates products will attempt to form a comparison degradation basis between ambient extended storage and accelerated oxidation under controlled laboratory conditions. The BioMates products accelerated ageing tests were performed at CERTH employing the fuels thermal-ageing apparatus that enables accelerated and prolonged ageing of conventional fuels, biofuels and their blends via heating and/or air contact. The apparatus functions under constant temperature ($T_{max}$ = 150°C), while the liquid volume capacity is 1 L (Figure 4) and enables the monitoring of temperature and/or air effect as well as the effect of metal contaminants.

Figure 4: Fuels thermal ageing apparatus of CERTH

In order to identify the optimal accelerated conditions, preliminary experiments with bio-based intermediates ("BioMates") were conducted exploring the temperature and air effect. The BioMates-material subjected to the accelerated ageing experiments was straw-based bio-oil from straight-run ablative fast pyrolysis bio-oil[1, p.6], upgraded via catalytic hydrotreatment at CERTH. More specifically, the BioMates products were accelerated aged at 60°C and 80°C for 6 h with and without air effect (100ml/min) for 6 h. Volumes of the samples were measured before and after ageing to ensure no loss of volatiles. After that, the samples were first analysed for their homogeneities to see whether phase separation had occurred or not and then the WC was measured. In all experiments, the volume loss of the aged BioMates products was less than 3% v/v, while phase separation was not noticed. Figure 5 presents the difference of the WC of the samples before and after their accelerated oxidation (ΔWC/Δt expressed in mg/kg/h).

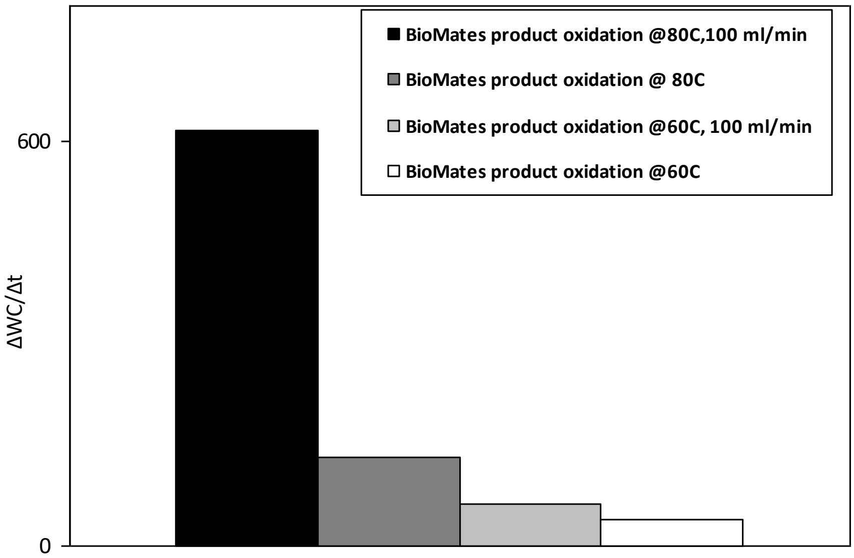


Figure 5: ΔWC/Δt of the BioMates products at different accelerating conditions

Based on these preliminary results the higher ΔWC/Δt was observed for the BioMates products acceleratedly oxidised at 80°C with 100 ml/min air effect, while the lowest ΔWC/Δt for the conditions involving only heating at 60°C. The WC increase could be an evidence of polymerization reactions (i.e. esterification) during the accelerated oxidation of BioMates products. The conditions identified for the BioMates products accelerated ageing involve heating at 80°C for 6 h, based on the WC increase of the oxidized samples compared to the WC increase of the samples that were stored for 12 months. Therefore, these accelerated conditions could be applied in an attempt to simulate BioMates product degradation after one year at ambient storage conditions. Furthermore, these identified accelerated conditions were employed for the investigation of the effect of the metal contaminant (stainless steel, dimensions: 6.5x2.5 cm, Figure 6) on the BioMates product stability. The results are presented in Figure 7.

Figure 6: Metal part added in the BioMates products for the accelerated experiments

These results show also the addition of a metal part resulted also in a considerable ΔWC/Δt increase of the BioMates products.

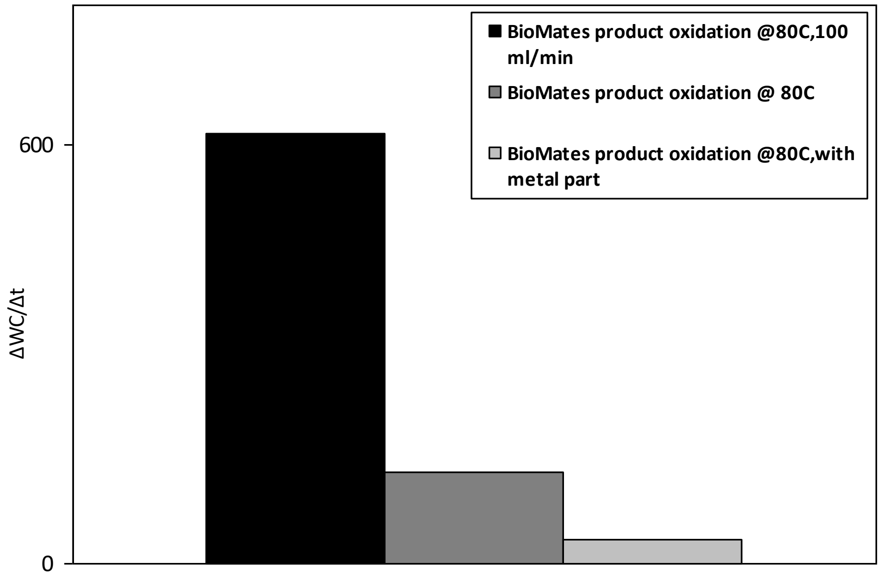


Figure 7: ΔWC/Δt of the BioMates products after accelerated oxidation at 80°C with different conditions

## 7. Conclusions

Energy integration is an essential piece of focus for realisation of new processes, as reduced net energy consumption by energy integration helps to improve efficiency and potentially even the economics. To assess the individual energy consumption/surplus of each process unit, detailed mass flow data and the specific properties of any streams going in and out of each process unit are need to be known for a proper assessment.

Another objective of this deliverable is to report on the BioMates product stabilization and its storage guidelines. Based on the presented results of the 12-month extended storage study, the proposed storage guidelines involve a maximum storage period of 5 months in ambient temperature without exposure to sunlight. It should be noted that an essential prerequisite is to systematically monitor the BioMates products quality variation (WC analysis is proposed) during their storage and also consider thoroughly other properties at the beginning of the storage period (i.e. TAN, viscosity, density etc.). Furthermore, taking into account the results of the accelerated ageing tests of the BioMates products, the accelerated ageing at 80°C for a period of 6 h resembles their quality deterioration after about 5 months of storage at ambient temperature. The WC increase of the accelerated aged BioMates products reveals potentially ageing reactions (i.e. esterification). The other accelerated conditions investigated involving air effect and metal contaminant result to more intense oxidation of the BioMates products, based on the WC evolution.

## 8. Disclaimer

This Deliverable report reflects only the authors' view; the European Commission and its responsible executive agency INEA are not responsible for any use that may be made of the information it contains.

## 9. Abbreviations

| | |
|---|---|
| CDU | Crude Distillation Unit |
| GHG | Greenhouse Gases |
| HDT | Hydrotreater |
| TAN | Total Acid Number |
| WC | Water Content |

## 10. Literature

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