Thermo-Catalytic Reforming of sewage sludge and hydrogenation of resulting TCR®-oil – A route to renewable chemicals and fuels

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e-EUBCE 2020
28th European Biomass Conference & Exhibition

E-EUBCE: 28th European Biomass Conference & Exhibition

July 06 – 09, 2020 Virtual

Jan Grunwald

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From solid waste Biomass to Bio-fuels

Analysis of the products – TCR®-oil

High quality, engine-ready

LHV: ≈34 MJ/kg

C	76.6 wt. %
H	9.0 wt. %
N	7.3 wt. %
S	1.6 wt. %
O (diff.)	3.5 wt. %
H_2O	2.0 wt. %
TAN	0.6 mg KOH/g
Ash	< 0.005 wt.%

- Thermal stable
- Low in O;S;N
- Low water content
- High heating value

→ **Excellent Precursor for Hydrogenation**

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From solid waste Biomass to Bio-fuels
Analysis of the products – TCR®-gas

Engine-ready gas

HHV:
≈14-18 MJ/m³

H_2	38 ± 3 v/v%
CO	8 ± 2 v/v%
CO_2	30 ± 3 v/v%
CH_4	14 ± 2 v/v%
C_xH_y	3 ± 1 v/v%

- **Required hydrogen for the hydrogenation can be used from TCR®-gas**
- **No further hydrogen source is needed**

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Product application – TCR®-oil and hydrogenated TCR®-oil

TCR®-oil

CHP Engine

Dual Fuel Engine (with Syngas)

Fuel Blends

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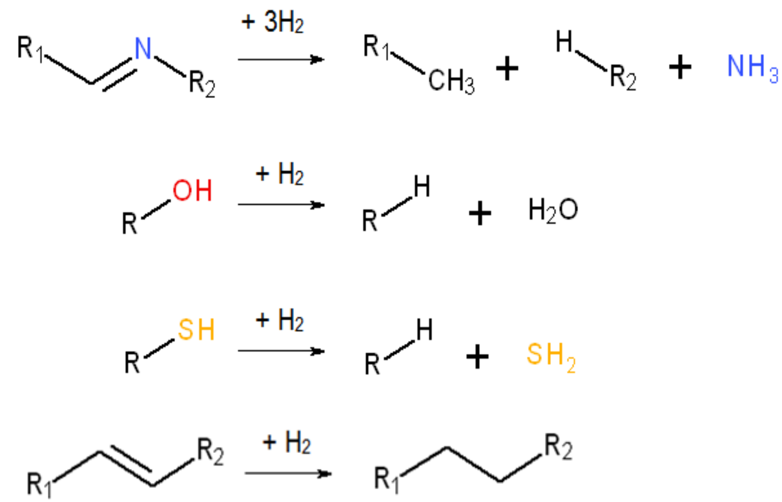
Upgrading of TCR®-oil - Catalytic hydrogenation

- Removal of N, O, S, Water
  - C & H content increase
- Saturation of double bounds
- NiMo or CoMo on Al_2O_3
- T = 240 - 400°C
- p = 40 - 150 bar
- Hydrogen consumption: 34.4 mmol H_2/g_{oil}



$$C_NH_MN_XO_YS_Z + K\ H_2 \rightarrow N\ CH_2 + X\ NH_3 + Y\ H_2O + Z\ H_2S$$

$$K = N + 1{,}5\ X + Y + Z - 0{,}5\ M$$

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From solid waste Biomass to Bio-fuels

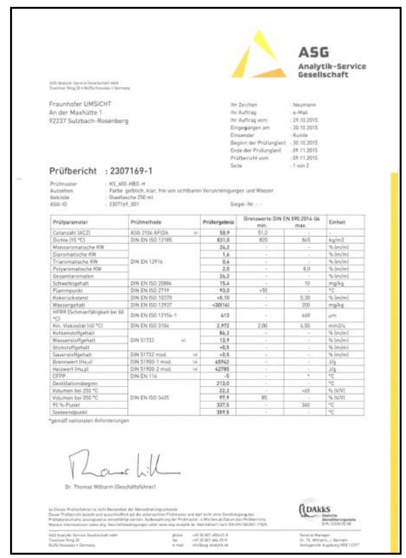
Product application – TCR®-oil and hydrogenated TCR®-oil

TCR®-oil

- CHP Engine
- Dual Fuel Engine (with Syngas)
- Fuel Blends

hydrogenated TCR®-oil

- Green Diesel
- Green crude Naphtha
- Green Chemicals



ASG Analytik-Service Gesellschaft

Fraunhofer UMSICHT
An der Maxhütte 1
92237 Sulzbach-Rosenberg

Ihr Zeichen : Neumann
Ihr Auftrag : e-Mail
Ihr Auftrag vom : 29.10.2015
Eingegangen am : 30.10.2015
Einsender : Kunde
Beginn der Prüfung(en) : 30.10.2015
Ende der Prüfung(en) : 09.11.2015
Prüfbericht vom : 09.11.2015
Seite : 1 von 2

Prüfbericht : 2307169-1

Prüfmuster : KS_600-HBO-H
Aussehen : Farbe: gelblich, klar, frei von sichtbaren Verunreinigungen und Wasser
Gebinde : Glasflasche 250 ml
ASG-ID : 2307169_001
Siegel-Nr. : -

Prüfparameter	Prüfmethode	Prüfergebnis	Grenzwerte DIN EN 590:2014-04 min.	max.	Einheit
Cetanzahl (ACZ)	ASG 2104 AFIDA (a)	58,9	51,0	-	-
Dichte (15 °C)	DIN EN ISO 12185	831,0	820	845	kg/m3
Monoaromatische KW	DIN EN 12916	24,2	-	-	% (m/m)
Diaromatische KW		1,6	-	-	% (m/m)
Triaromatische KW		0,4	-	-	% (m/m)
Polyaromatische KW		2,0	-	8,0	% (m/m)
Gesamtaromaten		26,2	-	-	% (m/m)
Schwefelgehalt	DIN EN ISO 20884	15,4	-	10	mg/kg
Flammpunkt	DIN EN ISO 2719	93,0	>55	-	°C
Koksrückstand	DIN EN ISO 10370	<0,10	-	0,30	% (m/m)
Wassergehalt	DIN EN ISO 12937	<30(16)	-	200	mg/kg
HFRR (Schmierfähigkeit bei 60 °C)	DIN EN ISO 12156-1	413	-	460	µm
Kin. Viskosität (40 °C)	DIN EN ISO 3104	2,972	2,00	4,50	mm2/s
Kohlenstoffgehalt	DIN 51732 (a)	86,2	-	-	% (m/m)
Wasserstoffgehalt		13,9	-	-	% (m/m)
Stickstoffgehalt		<0,5	-	-	% (m/m)
Sauerstoffgehalt	DIN 51732 mod. (a)	<0,5	-	-	% (m/m)
Brennwert (Ho,v)	DIN 51900-1 mod. (a)	45942	-	-	J/g
Heizwert (Hu,p)	DIN 51900-2 mod. (a)	42785	-	-	J/g
CFPP	DIN EN 116	-5	-	*	°C
Destillationsbeginn	DIN EN ISO 3405	213,0	-	-	°C
Volumen bei 250 °C		22,2	-	<65	% (V/V)
Volumen bei 350 °C		97,9	85	-	% (V/V)
95 %-Punkt		337,5	-	360	°C
Siedeendpunkt		359,5	-	-	°C

*gemäß nationalen Anforderungen

Dr. Thomas Wilharm (Geschäftsführer)

DAkkS Deutsche Akkreditierungsstelle

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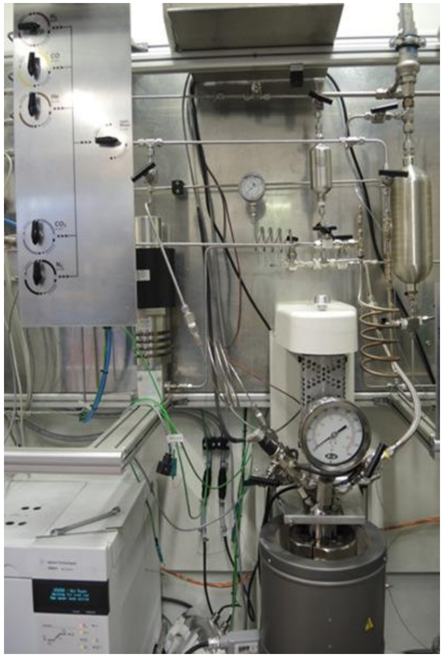
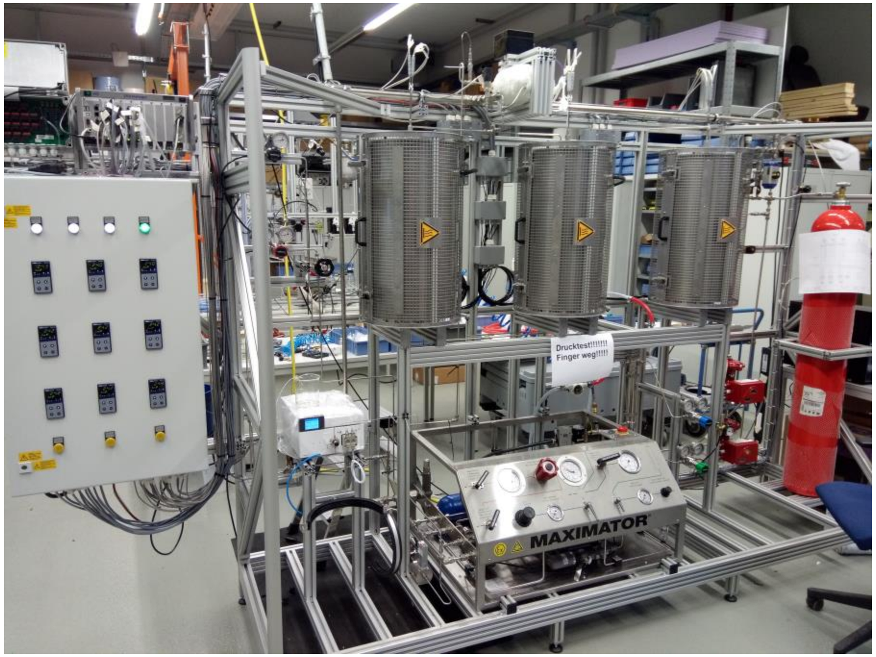
From solid waste Biomass to Bio-fuels

Hydrogenation plant – Batch and continuous plant



Batch-Reactor

- 500 ml reactor volume
- 30 ml sample volume



Continuous Reactor system

- 3 reactors with volume of 2.5 liter per reactor
- 3 kg/h feedstock input
- Gas recycling, GC online analysis

From solid waste Biomass to Bio-fuels

Analysis of TCR®-oil and hydrogenated TCR®-oil made of sewage sludge

TCR®-OIL

C	**76.6 wt%**
H	**9.0 wt%**
N	**7.3 wt%**
S	**1.6 wt%**
O (diff.)	**3.5 wt%**
H_2O	**2.0 wt%**
Ash	< 0.005 wt%

LHV	**34.8 MJ/kg**
TAN	**4.2 mg KOH/g**
Viscosity	9,1 mm²/s
Density	960 kg/m³

HYDROGENATION

HYDROGENATED TCR®-OIL

C	**86.2 wt%**
H	**13.8 wt%**
N	**< 0.1 wt%**
S	**0.0015 wt%**
O (diff.)	**< 0.1 wt%**
H_2O	**0.0016 wt%**
Ash	< 0.005 wt%

LHV	**42.8 MJ/kg**
TAN	**< 0.1 mg KOH/g**
Copper corr.	Grade 1
Flash point	< - 20 °C
Yield	**~ 80 %**

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GC-analysis off-gas of the hydrogenation step

Species	Amount	Units [vol.-% /ppm]
H_2	96.01	vol.-%
NH_3	2.1	vol.-%
H_2S	1.0	vol.-%
CO	0.01	vol.-%
CO_2	0.01	vol.-%
C_1-C_5	1.76	vol.-%

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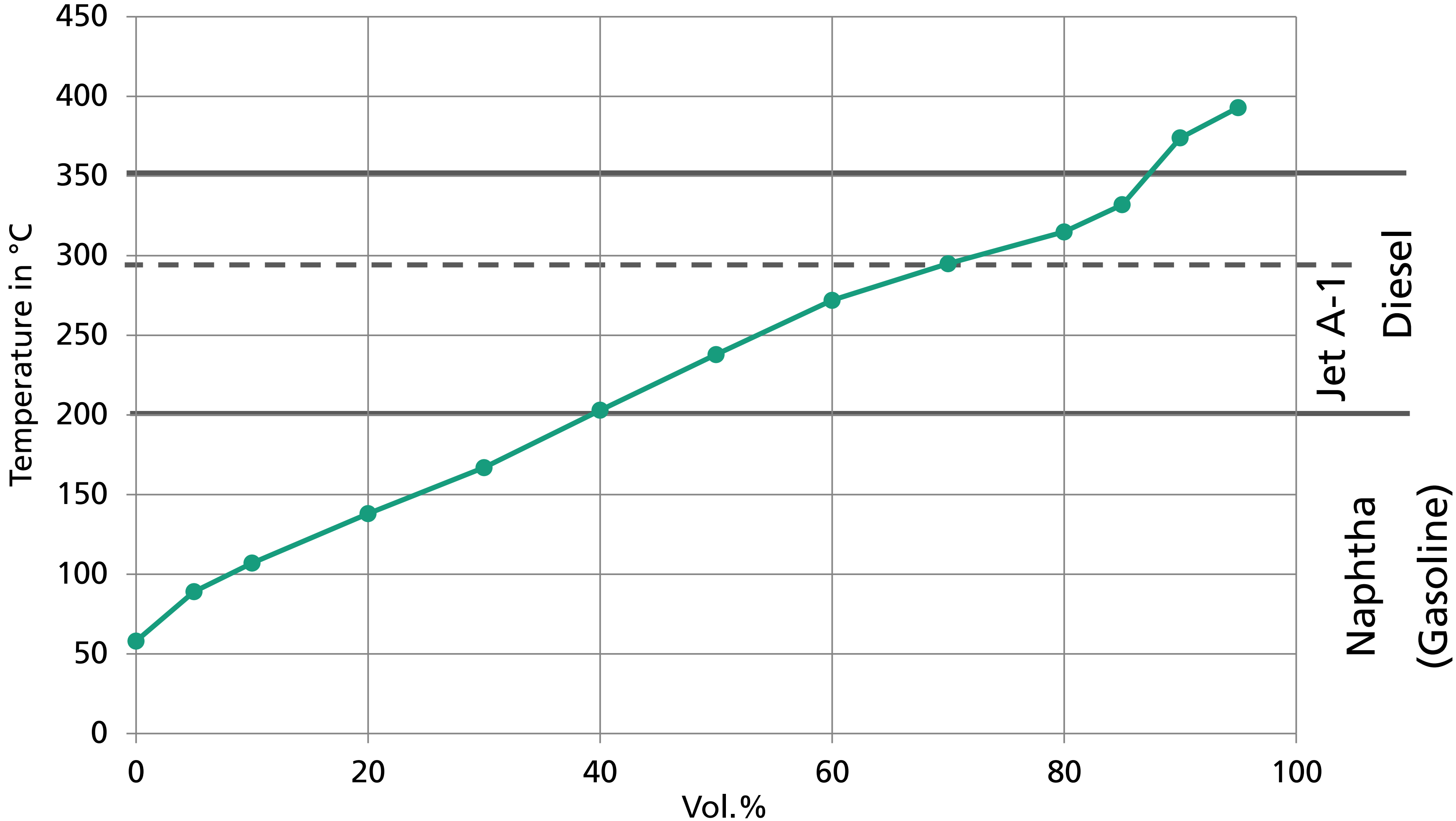
From solid waste Biomass to Bio-fuels
Distillation of hydrogenated TCR®-oil sewage sludge



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From solid waste Biomass to Bio-fuels

Comparison of the diesel fraction with EN 590

Test parameters	Test result	DIN EN 590 :2014	Unit
Cetane number (ICZ)	49,9 *	≥ 51	--
Cetane index (including density and distillation)	48,5	≥ 46	--
Density at 15 °C	824	820 - 845	kg/m^3
Polycyclic aromatic hydrocarbons	1,7	≤ 8,0	% (m/m)
Sulphur content	19,1 *	≤ 10,0	mg/kg
Flash point Pensky-Martens	69	> 55	°C
Coke residue (from 10% dest. residue)	< 0,10	≤ 0,30	% (m/m)
Ash content (775 °C)	< 0,001	≤ 0,01	% (m/m)
water content	101	≤ 200	mg/kg
Total Pollution	< 12	≤ 24	mg/kg
Corrosion effect on copper (3 h, 50 °C)	1	1	Korr.Grad
Fatty acid methyl ester content	< 0,1	≤ 7,0	% (V/V)
Oxidation stability (DIN EN ISO 12156-1 :2016)	5	≤ 25	g/m^3
Oxidation stability (DIN EN 15751 :2014)	> 48	≥ 20	h
HFRR (lubricity) at 60 °C	320	≤ 460	µm
Kinematic viscosity at 40 °C	1,861 *	2,00 - 4,50	mm^2/s
% (V/V) collected at 250 °C	66,7 *	≤ 65	% (V/V)
% (V/V) collected at 350 °C	> 98	≥ 85	% (V/V)
95 % (V/V) collected from	295,9	≤ 360	°C
CFPP / melting point (16.11-28.02)	-28	-20	°C
CFPP / melting point (01.10-15.11)	-28	-10	°C
CFPP / melting point (15.04-30.09)	-28	0	°C
Manganese content	< 0,5	≤ 2	mg/l
* Achievable by an adjustment of the hydrogenation			

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From solid waste Biomass to Bio-fuels

Comparison of the crude gasoline fraction with EN 228

Test parameters	Test result	DIN EN 228 :2014	Unit
ROZ	61 *	≥ 95	--
MOZ	< 60 *	≥ 85	--
Lead content	--	≤ 5,0	mg/kg
Density [15 °C]	772,9	720,0 - 775,0	kg/m³
sulfur content	5,5	≤ 10,0	mg/kg
Manganese	< 0,5	≤ 2,0	mg/l
Oxidation stability	> 360	≥ 360	min
Evaporation residue	< 0,5	≤ 5,0	mg/100 ml
Corrosion effect on copper	1	Klasse 1	Korr.Grad
Olefin content	0,54	≤ 18,0	% (V/V)
aromatics content	21,66	≤ 35,0	% (V/V)
benzene content	0,52	≤ 1,0	% (V/V)
Total oxygen OK	< 0,01	≤ 2,7	% (m/m)
methanol	< 0,01	≤ 3,0	% (V/V)
ethanol	< 0,01	≤ 5,0	% (V/V)
Isopropanol	< 0,01	up to max. 2.7% (m/m) Total oxygen content	% (V/V)
isobutanol	< 0,01		% (V/V)
tert-butanol	< 0,01		% (V/V)
ether (≥ 5 C-Atoms)	< 0,01		% (V/V)
Other oxygenated compounds	< 0,01		% (V/V)
Vapour pressure DVPE	9,3	45/60 - 60/90	kPa
Start of distillation	94,3	--	°C
Evaporated quantity at 70 °C	0 **	20/22 - 48/50	% (V/V)
Evaporated quantity at 100 °C	0,8 **	46/46 - 71/71	% (V/V)
Evaporated quantity at 150 °C	84,7	≥ 75/75	% (V/V)
Boiling end point	190,5	≤ 210,0	°C
Distillation residue	1	≤ 2,0	% (V/V)

* Achievable by further Catalytic reforming during the raffinery process

** Lack of light boilers due to laboratory distillation without cryocooler

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Conclusion

- Crude TCR®-oil highlights
  - Thermally stable (atmospheric distillation)
  - Directly suitable for hydrogenation
  - Required hydrogen can be used from TCR®-gas
- Hydrogenation highlights
  - Renewable chemicals extractable
  - Renewable crude gasoline and diesel (EN 590)
  - TCR® / Hydrogenation demonstration EU-project: To-Syn-Fuel

Thermo-Catalytic Reforming of sewage sludge and hydrogenation of resulting TCR®-oils – A route to renewable chemicals and fuels

Thank you for your Attention

Contact
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