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Tampere University

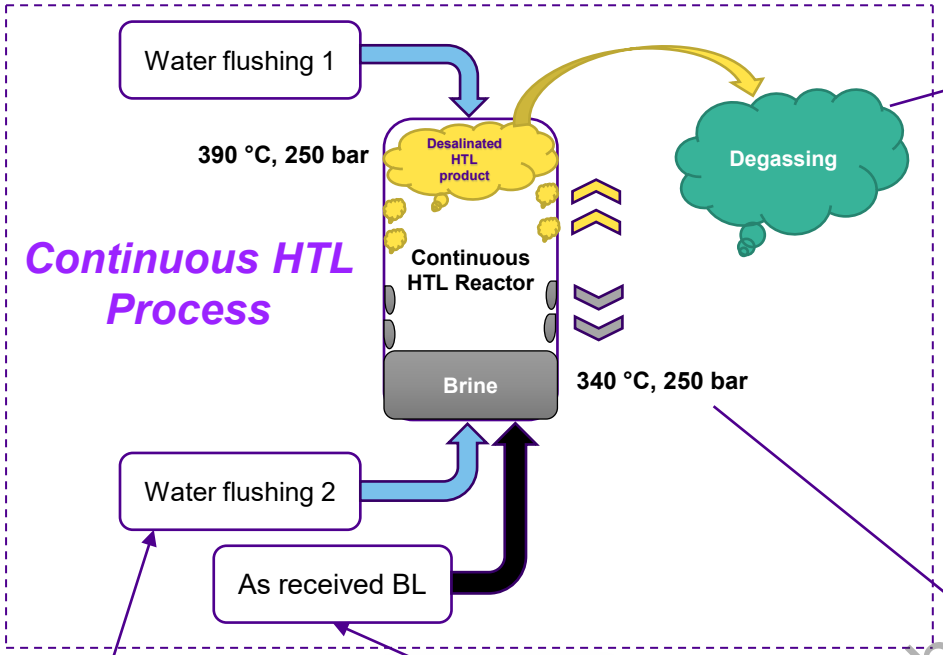
Meeting Concerning The Separation of HTL Products and Analysis

31 Jan 2023

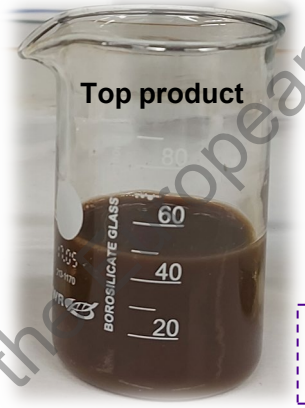
Separation method and first findings

Babak Arjmand

pH and Conductivity of Feed stream, Brine, & HTL product



Coolers for Top product



It is worth mentioning after diluting BL with Flushing water the pH and Conductivity are as follows:

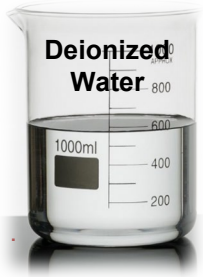
pH: 10,38

Conductivity: 36,5mS/cm

Top Product: pH = 8,56 ; Conductivity = 5.46 mS/cm

Deionized Water :
Black Liquor: pH = 6,52 ;

Conductivity = 5,35 µS/cm



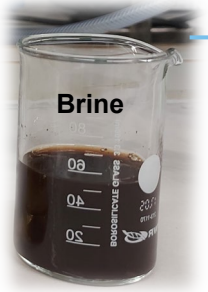
Black Liquor: pH = 13,4 ;

Conductivity = 68,9 mS/cm

Coolers for Bottom product (Brine)

Brine: pH = 9,37 ; Conductivity = 54,7 mS/cm

HTL Products Separation Process



Brine

Centrifuge (4500 rpm, 10 min)

HCL

Ethyl acetate

Effluent

Centrifuge (4500 rpm, 10 min)

Biocrude

Brine

Water

MEK

0.5 M citric acid

Solid Emulsion

Centrifuge (4500 rpm, 10 min)

Solid phase (Hydrochar)

Acetone washing

Filtration

Pure Hydochar

Liquid phase

Decantation

Biocrude + MEK

Rotary Evaporation

MEK

Pure Biocrude

Desalinated Top product

HCL

Ethyl acetate

Vacuum filteration

Acetone

Hydochar

Filtration

Pure Hydochar

Separatory funnel

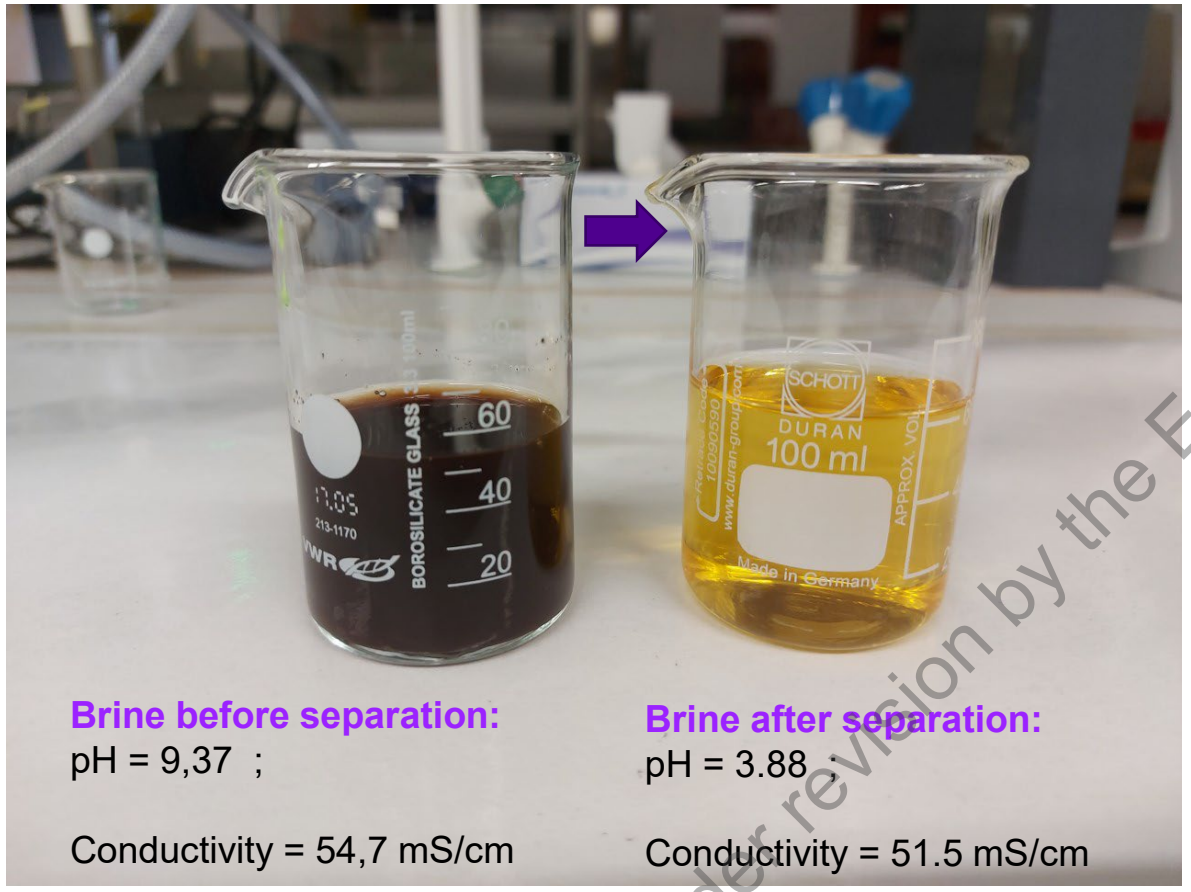
Aqueous phase

Biocrude + Ethyl acetate

Rotary Evaporation

Pure Biocrude

Final products after acidification and separation



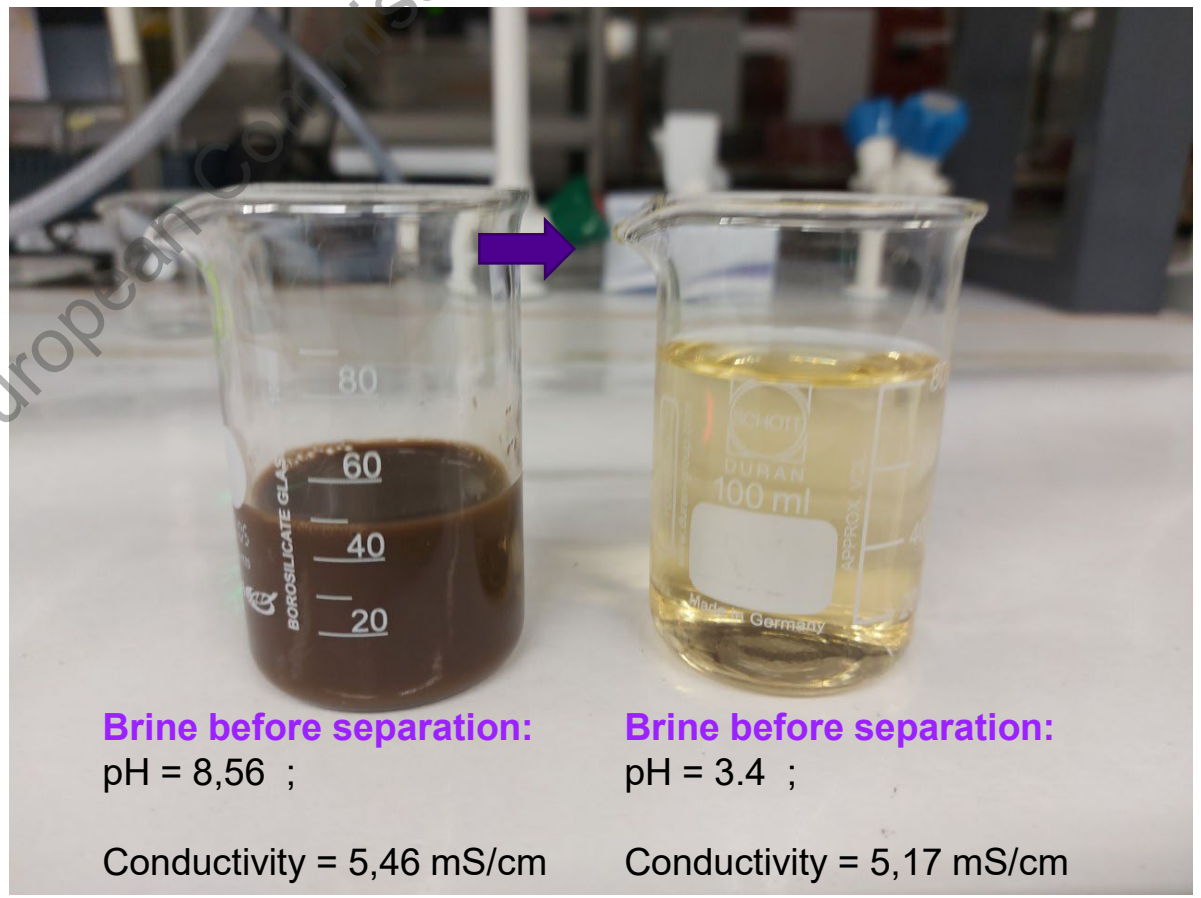
Brine before separation:
pH = 9,37 ;

Conductivity = 54,7 mS/cm

Brine after separation:
pH = 3.88 ;

Conductivity = 51.5 mS/cm

TOC = 10300 mg/L



Brine before separation:
pH = 8,56 ;

Conductivity = 5,46 mS/cm

Brine before separation:
pH = 3.4 ;

Conductivity = 5,17 mS/cm

TOC= 1289 mg/L

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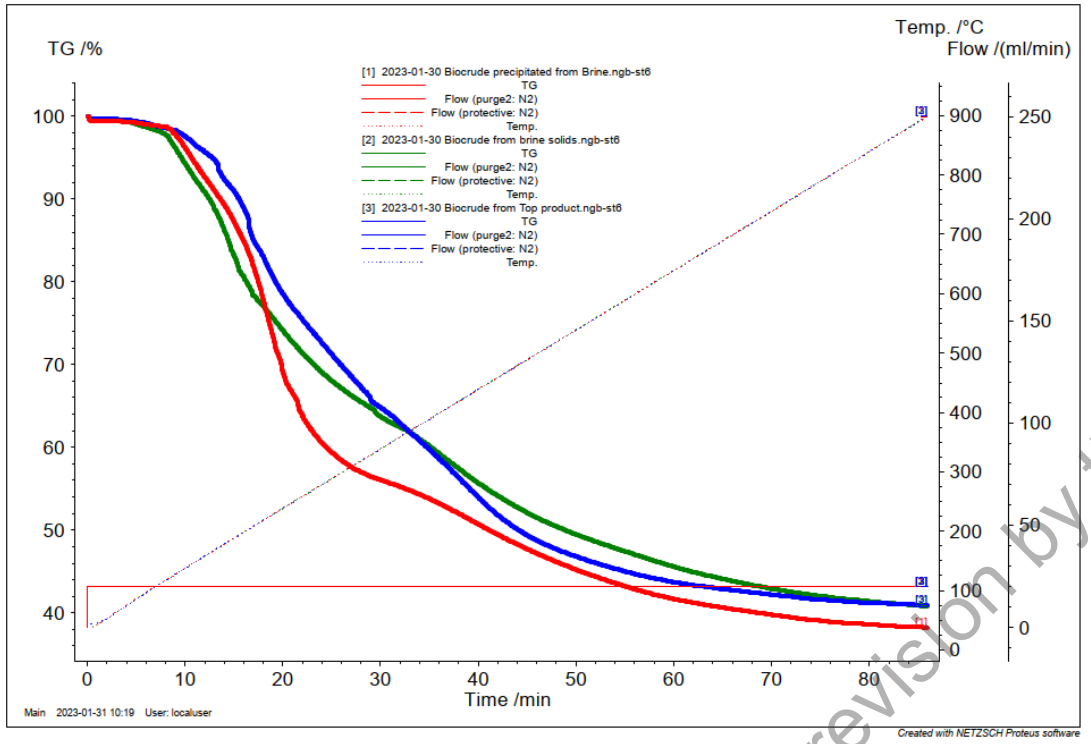
Elemental Analysis

Sample	C	H	N	S	O	Ash	HHV
Lignin	57	5.21	0.1122	21	34	1.58	29.90184
DBL	32	3	0.0142	0.6824	19	45.04	17.064
Hydrochar from Brine Solids	73.9312	3.3737	0.79743	0	16.6847	5.213	31.81569
Hydrochar Precipitated from Brine	54.853	2.89314	1.18643	0	37.6374	3.43	26.89737
Hydrochar Top Product	76.1597	4.48894	0.81464	0	16.1487	2.388	33.83708
Biocrude Top Product	75.3649	5.93507	0.50289	0.85886	17.3382	0	35.35075
Biocrude from Brine Solids	64.7283	6.46386	0.2978	0	28.5101	0	33.45155
Biocrude Precipitated from Brine	63.586	4.94743	0.47803	3.31839	27.6701	0	31.20564

Thermogravimethric Analysis



	Moisture	VM	FC
Biocrude from Top product	2,2	56,61	41,187
Biocrude from Brine solids	3	54,517	42,483
Biocrude precipitated from Brine	2	59.205	38.794

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Pyro-GC-MS Qualitative Analysis of Biocrude from Brine Solids

1	2,5-Furandione, 3-methyl-
2	2-Cyclopenten-1-one, 2,3-dimethyl-
3	1-Ethylpentyl acetate
4	2-Cyclopenten-1-one, 3,4,4-trimethyl-
5	p-Cresol
6	Ethanone, 1-(1-cyclohexen-1-yl)-
7	2-Cyclopenten-1-one, 2,3,4,5-tetramethyl-
8	Ethanone, 1-(3-methylphenyl)-
9	Phenol, 2,3-dimethyl-
10	2-Cyclopenten-1-one, 2,3,4,5-tetramethyl-
11	Phenol, 3-ethyl-
12	1H-Indene, 2,3-dihydro-1,6-dimethyl-
13	Phenol, 3,4-dimethyl-
14	Catechol
15	Benzene, 1-ethyl-4-methoxy-
16	Ethanone, 1-(2,5-dimethylphenyl)-
17	Ethanone, 1-(2,4-dimethylphenyl)-
18	Phenol, 3-ethyl-5-methyl-
19	1,2-Benzenediol, 4-methyl-
20	4-Methoxy-2,6-dimethylphenol
21	1H-Inden-1-one, 2,3-dihydro-
22	1,2-Benzenediol, 4-methyl-
23	1,3-Benzenediol, 2,5-dimethyl-
24	1-Methylindan-2-one
25	Benzene, 1-(1-methylethenyl)-3-(1-methylethyl)-
26	1,3-Benzenediol, 2,5-dimethyl-
27	Phenol, 2-(2-methyl-2-propenyl)-
28	(1-Methylpenta-1,3-dienyl)benzene
29	Ethyl p-hydroxybenzoate
30	7-Methylindan-1-one
31	1,3-Benzenediol, 4,5-dimethyl-
32	Benzene, 1,4-dimethoxy-2-methyl-
33	1,4-Benzenediol, 2,3,5-trimethyl-
34	3-Isopropyl-1,2-benzenediol
35	Benzene, hexamethyl-
36	1,3-Benzenediol, 4-propyl-
37	Benzene, 1-methoxy-4-(1-methyl-2-propenyl)-
38	3-Isopropyl-1,2-benzenediol
39	4-Methoxy-2,6-dimethylphenol
40	1-Naphthalenol
41	1,4-benzenediol, 2-(1-methylpropyl)-
42	4-(Cyclopent-2-en-1-yl)phenol
43	Benzene, 1,3,5-trimethyl-2-(1-methylethenyl)-
44	Benzene, 3-ethyl-1,2,4,5-tetramethyl-
45	1-Hydroxy-1,2,3,4-tetrahydronaphthalene-8-carboxaldehyde
46	1,4-benzenediol, 2-(1-methylpropyl)-
47	2,3-2H-Benzofuran-2-one, 3,3,4,6-tetramethyl-
48	1-Naphthalenol, 2-methyl-
49	1-Methyl-2-naphthol
50	7-Methylnaphthalen-2-ol
51	7-Methyl-1-naphthol
52	1-Naphthalenol, 2-methyl-
53	Benzene, 1,2,3,4-tetramethyl-4-(1-methylethenyl)-
54	1H-Inden-1-one, 2,3-dihydro-3,4,7-trimethyl-
57	1-Naphthol, 6,7-dimethyl-
62	1-Naphthol, 5,7-dimethyl-
64	1-Naphthol, 2,5,8-trimethyl-
66	Chamazulene
67	Naphthalene, 1,6-dimethyl-4-(1-methylethyl)-
68	Benzenamine, 4,4'-methylenebis[2-methyl-
69	5(10H)-Pyrido[3,4-b]quinolone, 7-methoxy-
70	Tetrapentacontane, 1,54-dibromo-
72	17-Pentatriacontene
73	Tetrapentacontane, 1,54-dibromo-
74	Dotriacontyl pentafluoropropionate
75	.gamma.-Sitosterol
76	2-Methylhexacosane

40 to 400 °C

1	Propane
2	2-Butene, (E)-
3	2-Hexanone, 5-methyl-
4	1,3-Pentadiene, (E)-
5	1,3-Cyclopentadiene
6	1,3-Cyclohexadiene
7	Benzene
8	Thiophene
9	Toluene
10	Thiophene, 3-methyl-
11	p-Xylene
12	p-Xylene
13	Phosphonic acid, (p-hydroxyphenyl)-
14	Mesitylene
15	Indene
16	Phenol, 2-methyl-
17	Phenol, 2-methyl-
18	2-Methylindene
19	Phenol, 3,4-dimethyl-
20	Naphthalene
21	Phenol, 3,4-dimethyl-
22	Catechol
23	Phenol, 2-ethyl-4-methyl-
24	1,2-Benzenediol, 4-methyl-
25	
26	Naphthalene, 2-methyl-
27	Naphthalene, 2-methyl-
28	4,7-Methano-1H-indene-1,8-dione, 3a,4,7,7a-tetrahydro-
29	Benzene, 2-ethenyl-1,3,5-trimethyl-

400 to 850 °C

Pyro-GC-MS Qualitative Analysis of Precipitated Biocrude from Brine

1	Dimethyl trisulfide
2	Phenol, 2-methyl-
3	2-Cyclopenten-1-one, 3,4,4-trimethyl-
4	Phenol, 3-methyl-
5	Ethanone, 1-(1-cyclohexen-1-yl)-
6	Phenol, 3,4-dimethyl-
7	Phenol, 3,4-dimethyl-
8	Phenol, 2,5-dimethyl-
9	Phenol, 3,4-dimethyl-
10	Catechol
11	Tetrasulfide, dimethyl
12	Phenol, 3-(1-methylethyl)-
13	Phenol, 2,3,5-trimethyl-
14	Phenol, 2-ethyl-4-methyl-
15	1,3-Cyclopentadiene, 1,2,3,4,5-pentamethyl-
16	Phenol, 3-ethyl-5-methyl-
17	1,2-Benzenediol, 4-methyl-
18	Phenol, 2,3,5-trimethyl-
19	4-Methoxy-2,6-dimethylphenol
20	1,3-Benzenediol, 2-methyl-
21	1,3-Benzenediol, 2,5-dimethyl-
22	Ethyl p-hydroxybenzoate
23	2,3-Dimethylhydroquinone
24	4-Hydroxy-1-indanone
25	2,3-Dimethylhydroquinone
26	1,3-Benzenediol, 4,5-dimethyl-
27	1,4-Benzenediol, 2,5-dimethyl-
28	4-Methoxy-2,6-dimethylphenol
29	1,4-Benzenediol, 2,3,5-trimethyl-
30	4-Methoxy-2,6-dimethylphenol
32	Benzaldehyde, 2,3,4,5-tetramethyl-
33	3-Isopropyl-1,2-benzenediol
34	1,3-Benzenediol, 4-propyl-
35	Benzaldehyde, 2,3,4,5-tetramethyl-
36	3-Isopropyl-1,2-benzenediol
37	Benzofuran, 2,3-dihydro-2,2,4,6-tetramethyl-
38	4-Methoxy-2,6-dimethylphenol
39	1-Naphthalenol
40	2',4'-Dihydroxypropiophenone
41	1,3,5-Cycloheptatriene, 2,5-diethyl-7,7-dimethyl-
42	1H-Inden-1-one, 2,3-dihydro-3,3-dimethyl-
45	2',4'-Dihydroxypropiophenone
47	p-Cymene-2,5-diol
49	Phenol, 2-ethoxy-4-(2-propenyl)-
50	1-Naphthalenol, 2-methyl-
51	1-Methyl-2-naphthol
52	7-Methylnaphthalen-2-ol
61	1-Naphthol, 6,7-dimethyl-
62	1H-Inden-1-one, 2,3-dihydro-7-hydroxy-3-methyl-
63	5-Hydroxy-3-methyl-1-indanone
64	1,5-Naphthalenediol
65	2H-1-Benzopyran-2-one, 3,4-dihydro-4,4-dimethyl-
68	1-Naphthol, 6,7-dimethyl-
70	1H-Inden-1-one, 2,3-dihydro-5-methoxy-
71	Benzofuran, 5-methoxy-6,7-dimethyl-
72	Benzofuran, 2,3-dihydro-2,2,4,6-tetramethyl-
73	1,4-Methanonaphthalene-5,8-diol, 1,2,3,4-tetrahydro-
74	2H-1-benzopyran-2-one, 3,4-dihydro-4,4,6-trimethyl-
76	2,2'-Ethylidenebis(5-methylfuran)
79	3-Methylindene-2-carboxylic acid
85	2H-1-Benzopyran-2-one 3,4-dimethyl-
86	Tetradecanoic acid
89	8-Methoxy-2,2,4-trimethyl-1,2-dihydroquinoline
91	Benzene, 1-(1-cyclohexen-1-yl)-4-methoxy-
94	6-Quinolinecarboxaldehyde, 1,2-dihydro-1,2,2,4-tetramethyl-
95	Thiazolo[4,5-f]quinoline, 7,9-dimethyl-
98	Bis(2-ethylhexyl) phthalate
99	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester

40 to 400 °C

1	o-Ethylhydroxylamine
2	Palladium, bis[(1,2,3-.eta.)-2-butenyl]-
3	2-Hexanone, 5-methyl-
4	1,3-Pentadiene, (E)-
5	1,3-Cyclopentadiene
6	1-Penten-3-yne
7	2-Butanone
8	1,3-Butadiene, 2,3-dimethyl-
9	1,4-Cyclohexadiene
10	Benzene
11	Thiophene
12	Toluene
13	Thiophene, 3-methyl-
14	p-Xylene
15	Styrene
16	p-Xylene
17	Benzene, 1-ethyl-2-methyl-
18	Mesitylene
19	Phosphonic acid, (p-hydroxyphenyl)-
20	Mesitylene
21	Benzofuran
22	Indene
23	Phenol, 2-methyl-
24	Phenol, 3-methyl-
25	Phenol, 3,4-dimethyl-
26	Benzene, 1-methyl-4-(1-propynyl)-
27	1H-Indene, 1-methyl-
28	Azulene
29	Phenol, 3,4-dimethyl-
30	Phenol, 2,3-dimethyl-
31	Naphthalene
32	Phenol, 3,4-dimethyl-
33	Catechol
34	Phenol, 2,4,6-trimethyl-
35	4-Vinylphenol
36	Phenol, 2-ethyl-4-methyl-
37	1,2-Benzenediol, 4-methyl-
38	Benzocycloheptatriene
39	Phenol, 2,4,6-trimethyl-
40	1,2-Benzenediol, 4-methyl-
41	Naphthalene, 2-methyl-
42	Naphthalene, 2-methyl-
43	Hydroxychavicol
44	5-Methyl-1-phenyl-1H-tetrazole
45	Naphthalene, 1,7-dimethyl-
46	Naphthalene, 1,4-dimethyl-
47	Biphenyl
48	Naphthalene, 2,3-dimethyl-
49	Biphenylene
50	Cyclopropane, 1-(1-hydroxyethyl)-2-methylene-1-phenyl-
51	Carbaril
52	1-Naphthalenol
53	Naphthalene, 1,4,6-trimethyl-
55	Fluorene
58	7-Methylnaphthalen-2-ol
62	1-Naphthol, 6,7-dimethyl-
65	Phenanthrene
66	Phenol, 2-(phenylmethyl)-
67	4,6,8-Trimethyl-1-azulenecarbaldehyde
68	4,6,8-Trimethyl-1-azulenecarbaldehyde
69	2-Hydroxyfluorene
70	2-Hydroxyfluorene
72	Naphtho[2,1-b]furan, 1,2-dimethyl-
74	Phenol, 4,4'-methylenebis-
77	3-Phenanthrol
80	Pyrene, 1,2,3,6,7,8-hexahydro-
82	.beta.-Carboline, 8-methoxy-1-methyl-
84	.beta.-Carboline, 8-methoxy-1-methyl-
86	Benzo[b]naphtho[2,3-d]furan
92	Indeno[2,1-b]chromene,

400 to 850 °C

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Pyro-GC-MS Qualitative Analysis of Top Product

1	2-Cyclopenten-1-one, 3,4,4-trimethyl-
2	2-Cyclopenten-1-one, 2,3,4,5-tetramethyl-
3	Phenol, 2,6-dimethyl-
4	Phenol, 2,4-dimethyl-
5	3,4,5,6,7,8-Hexahydro-2H-chromene
6	Phenol, 2,4,6-trimethyl-
7	Catechol
8	Ethanone, 1-(2,4-dimethylphenyl)-
9	1H-Indene, 1,3-dimethyl-
10	1,2-Benzenediol, 4-methyl-
11	1H-Inden-1-one, 2,3-dihydro-
12	1,2-Benzenediol, 4-methyl-
13	1H-Inden-1-one, 2,3-dihydro-2-methyl-
14	1,3-Benzenediol, 2,5-dimethyl-
15	1-Methylindan-2-one
16	1-Propanone, 1-(2,4-dimethylphenyl)-
17	Benzene, 1-(1-methylethenyl)-3-(1-methylethyl)-
18	1,3-Benzenediol, 2,5-dimethyl-
19	5,8-Dimethyl-1,2,3,4-tetrahydro-1-naphthol
20	Ethanone, 1-(3,4,5-trimethylphenyl)-
21	1,3-Benzenediol, 4,5-dimethyl-
22	7-Methylindan-1-one
23	1,3-Benzenediol, 4,5-dimethyl-
24	7-Methylindan-1-one
25	Naphthalene, 1,2,3,4-tetrahydro-6,7-dimethyl-
26	4-Methoxy-2,6-dimethylphenol
27	1,4-Benzenediol, 2,3,5-trimethyl-
28	3-Isopropyl-1,2-benzenediol
29	1H-Inden-1-one, 2,3-dihydro-7-hydroxy-3-methyl-
30	Phenol, 2-(2-penten-4-yl)-4-methyl-
31	Benzaldehyde, 2,3,4,5-tetramethyl-
32	(S)-1-(4-Methoxyphenyl)-2-methylpropyl acetate
33	Benzofuran, 2,3-dihydro-2,2,5,6-tetramethyl-
34	4-Methoxy-2,6-dimethylphenol
35	1-Naphthalenol
36	(E)-4-(3,4-Dimethoxyphenyl)but-3-en-1-yl acetate
37	1H-Inden-1-ol, 2,3-dihydro-3,3-dimethyl-
38	Benzene, 1,3,5-trimethyl-2-(1-methylethenyl)-
39	1-Hydroxy-1,2,3,4-tetrahydronaphthalene-8-carboxaldehyde
40	4-(Cyclopent-2-en-1-yl)phenol
41	Coumarin, 3,4-dihydro-4,5,7-trimethyl-
42	1-Methyl-2-naphthol
43	7-Methylnaphthalen-2-ol
44	1-Naphthalenol, 2-methyl-
45	Benzene, 1,2,3,4-tetramethyl-4-(1-methylethenyl)-
46	1-Naphthol, 6,7-dimethyl-
47	1-Naphthol, 5,7-dimethyl-
48	1-Naphthol, 2,5,8-trimethyl-
49	Azulene, 1,4-dimethyl-7-(1-methylethyl)-
50	2-Methylhexacosane
51	Carbonic acid, decyl heptadecyl ester
52	Hexacosyl nonyl ether
53	2-Methylpentacosane
54	Nonadecyl heptafluorobutyrate
55	Dotriacontyl pentafluoropropionate
56	Tetrapentacontane, 1,54-dibromo-
57	Octatriacontyl trifluoroacetate
58	Tetrapentacontane, 1,54-dibromo-
59	2-Methylhexacosane
60	Hexatriacontane
61	15-Methylnonacosane
62	Tetrapentacontane, 1,54-dibromo-
63	Carbonic acid, octadecyl vinyl ester

40 to 400 °C

1	Ethyl hydrogen oxalate
2	2-Butene, (E)-
3	2-Hexanone, 5-methyl-
4	1,3-Pentadiene
5	1,3-Cyclopentadiene
6	3-Hexene, (E)-
7	1,4-Cyclohexadiene
8	1,3-Cyclopentadiene, 1-methyl-
9	Benzene
10	1,3-Cyclopentadiene, 2-methyl-
11	Toluene
12	Ethylbenzene
13	p-Xylene
14	Styrene
15	Benzene, 1-ethyl-3-methyl-
16	Phosphonic acid, (p-hydroxyphenyl)-
17	Mesitylene
18	Indene
19	Phenol, 2-methyl-
20	2-Methylindene
21	Benzene, 1-methyl-4-(1-propynyl)-
22	Azulene
23	Phenol, 3,4-dimethyl-
24	Naphthalene
25	Phenol, 3,4-dimethyl-
26	Phenol, 3-ethyl-5-methyl-
27	Phenol, 2,3,5-trimethyl-
28	Naphthalene, 2-methyl-
29	Naphthalene, 1,3-dimethyl-
30	Biphenylene
31	Hexatriacontane
32	Tetrapentacontane
33	Hexatriacontane
34	Tetrapentacontane, 1,54-dibromo-
35	Dotriacontyl pentafluoropropionate
36	Tetrapentacontane, 1,54-dibromo-
37	2-Methylhexacosane
38	Tetrapentacontane, 1,54-dibromo-
39	Tetrapentacontane, 1,54-dibromo-
40	Heptacosyl heptafluorobutyrate
41	Tetrapentacontane, 1,54-dibromo-
42	Eicosyl trifluoroacetate
43	11-Methyltricosane
44	Hexatriacontane
45	2-Methylhexacosane

400 to 850 °C

Next experiments

1. Washing and extracting the remaining Biocrude and Hydrochar from the Reactor and Coolers.
2. Decreasing the Flushing rate (maybe 50/50), to investigate the influence of higher pH and conductivity on the final products
3. Using different solvents for extraction
4. Purging the cHTL process with CO2

Thanks for your time and attention!

Babak Arjmand