Data Sets: Material balance of hop extracts, HPLC analysis of hop extracts and GC-MS analysis of hop extracts

Thesis: Aroma and Bitter Extracts from South African Hops: Characterisation and Extraction Process Development using Supercritical CO_2

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Experiment: Trial run on Southern Star

Description: This run was on the 19th April 2021 at 07:14 am. A purge of the system was done and the pressure was set to 150 bar. A feed of 1118.53 g was loaded. At 11:05 am static extraction occurred for 30 minutes. At 11:40, the pressure was increased to 150 bar as some pressure was lost during static extraction and then dynamic extraction began. At 12:10 the first sample was collected with a mass of 39.91 g. Sampling occurred at a 30 minute interval. At the 1 hour mark an extract with a mass of 50.12 g was collected. After running for 90 min, the plant tripped and this feed was compromised. At this point a mass of 11.73 g was collected.

Parameters:

Temperature (°C)	40
Pressure (bar)	150
Solvent flowrate (kg/h)	6
Solvent density (kg/m^3)	780

Results:

Trial run 150 bar

Time (min)	Mass extract (g)	Mass without bottle (g)	Mass	Yield
30	283.58	39.91	39.91	0.036
60	333.7	90.03	50.12	0.045
90	345.43	101.76	11.73	0.011

Experiment: Southern Star

Description: This run was done on 20th April 2021. The hop type was Southern star with a mass of 1006 g of ground hops. The hops were loaded and pressure was increased to 150 bar. At 09:51 am, static extraction began for 30 minutes. At 10:24 am dynamic extraction began and the first sample was collected at 10:54 am with an extract mass of 39.32 g. Sampling occurred every 30 minutes for 2 hours.

The pressure was increased to 200 bar. A 30 minute sampling procedure was followed.

The pressure was increased to 250 bar. A 30 minute sampling procedure was followed At 15 min before reaching 120 min the plant tripped. The speculation was that due to long hours of operation the plant had an emergency shutdown as no error was visible from pilot plant data.

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Parameters:

Temperature (°C)	40
Pressure (bar)	150, 200, 250
Solvent flowrate (kg/h)	6
Solvent density (kg/m^3)	780, 840, 880

Run 1 data at 150bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
30	285.62	39.32	39.32	0.039
60	332.5	86.2	46.88	0.047
90	350.92	104.62	18.42	0.018
120	370.85	124.55	19.93	0.019

200bar

Time (min)	Mass extract (g)	Mass without bottle (g)	Mass	Yield
30	259.14	12.74	12.74	0.013
60	284.41	38.01	25.27	0.025
90	296.25	49.85	11.84	0.012
120	303.77	57.37	7.52	0.0075

250bar

Time (min)	Mass extract (g)	Mass without bottle (g)	Mass	Yield
30	254.3	7.47	7.47	0.0074
60	260.4	13.57	6.1	0.0061
90	270.47	23.64	10.07	0.01

Experiment: Southern Passion

Description: Due to the plant emergency shutdown, it was decided to complete the remaining hop types at 2 pressures for 2 hours. Run 2 was completed on the 28th April 2021. The hop type was Southern passion with a mass of 1014.44 g. The run began at 6:30 am. After grinding the hops, purging the system and loading the hop feed at 09:59 am. The pressure was increased to 200 bar for static extraction. During static extraction a pressure drop of at least 15 bar was noted during previous runs. Thus it was decided to set the pressure to 230 bar. Dynamic extraction began at 10:56 and at 11:56 the first sample was collected. In order to keep the plant at steady state, samples were now collected at a one hour interval. The pressure was increased to 250 bar for two hours.

Parameters:

Temperature (°C)	40
Pressure (bar)	200, 250
Solvent flowrate (kg/h)	6
Solvent density (kg/m^3)	840, 880

Run 2 data at 200bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	311.18	67.55	67.55	0.066
120	366.07	122.44	54.89	0.054

250bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	272.29	28.12	28.12	0.027
120	288.73	44.56	16.44	0.016

Experiment: African Queen

Description: This run was done on the 30th April 2021. The feed was African queen hops with a mass of 911.27 g. At 09:40 am the feed was loaded and static extraction began at 10:21. At 10:56 am dynamic extraction began and sampling followed after an hour.

The pressure was increased to 250 bar. For this pressure, an extraction time of three hours was tried.

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Parameters:

Temperature (°C)	40
Pressure (bar)	200, 250
Solvent flowrate (kg/h)	6
Solvent density (kg/m^3)	840, 880

Run 3 data at 200bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	322.73	78.83	78.83	0.087
120	364.38	120.48	41.65	0.046

250bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	258.16	15.11	15.11	0.017
120	273.24	30.19	15.08	0.017
180	282.07	39.02	8.83	0.0097

Experiment: Southern Promise

Description: This run was done on the 4th May 2021. This feed was Southern promise, with a mass of 1002.19 g. This hop type was grinded the Friday and loaded into the vessel. It was left isolated at a pressure of 8bar over the weekend. At 07:27 am static extraction began and by 08:01 dynamic extraction began. The pressure was increased to 250 bar. After 60 min and extract of 25.85 g was collected. The plant then tripped. It was speculated that the chiller may have a leak.

Parameters:

Temperature (°C)	40
Pressure (bar)	150, 250
Solvent flowrate (kg/h)	6
Solvent density (kg/m^3)	780, 880

Run 4 data at 150bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	341.53	93.87	93.87	0.094
120	363.59	115.93	22.06	0.022
180	380.02	132.36	16.43	0.016

250bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	273.14	25.85	25.85	0.026

Experiment: Southern star rerun

Description: This was a rerun of run 1. This feed was Southern star with a mass of 1002.45 g. At 07:55 am static extraction began. By 08:30 dynamic extraction started. This pressure was increased to 200 bar. This pressure was increased to 250 bar.

Parameters:

Temperature (°C)	40
Pressure (bar)	150, 200, 250
Solvent flowrate (kg/h)	6
Solvent density (kg/m^3)	780, 840, 880

Run 5 data at 150bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	316.79	73.08	73.08	0.072
120	369.96	126.25	53.17	0.053

200bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	272.05	27.55	27.55	0.027
120	308.94	64.44	36.89	0.036

250bar

Time (min)	**Mass extract (g)**	**Mass without bottle (g)**	**Mass**	**Yield**
60	260.09	19.62	19.62	0.019
120	281.38	40.91	21.29	0.021

HPLC analysis

Description: All HPLC analysis was carried out on a Column: YMC-Pack Pro C18 with the dimensions 150 mm x 4.6 mm, 5 µm. The system was equipped with a quaternary pump, a temperature-controlled column chamber, an autosampler and a degasser membrane. The method carried out follows the method in the article titled *—An improved HPLC method for single-run analysis of the spectrum of hop bittering compounds usually encountered in beers*‖ (Oladokun et al., 2016).

	Cohumulone	**N+ adhumulone**	**Colupulone**	**n+ adlupulone**
	mg/g	mg/g	mg/g	mg/g
Southern Star 250 bar @ 240min	16.62	44.27	3.55	3.46
Southern Promise 250bar	10.28	36.56	8.54	7.80
Southern Star 250bar @ 120min	14.84	37.56	8.83	8.80
Southern Star 200bar	14.39	36.71	9.49	9.18
Southern Star Rerun 200bar	14.70	41.38	9.43	9.11
Southern Star 250bar	17.78	45.24	6.53	4.42
Southern passion 250bar	14.70	46.38	7.86	9.93
Southern Star Rerun 250bar	16.38	43.39	7.78	7.58
African Queen 250bar	16.91	41.45	9.96	7.61
	mg/L	mg/L	mg/L	mg/L
Solvent Extraction Southern passion (wet hopping)	1.16	2.48	N.D.	N.D.
Solvent Extraction Southern passion (dry hopping)	0.44	1.86	N.D.	N.D.
Southern star 250bar +ethanol	N.D.	N.D.	N.D.	N.D.

GC-MS analysis

Description: The GC-MS analysis was done on a Capillary Column Model Number: Zebron 7HG-G007-11ZB-WAX. This column has a maximum temperature of 250°C with a nominal length of 30 m, a nominal diameter of 250 µm and a nominal film thickness of 0.25 µm.

GC method

For the GC method the oven had an initial temperature of 40°C and an initial time of 8 minutes. The run time was 38.57 minutes with a maximum temperature of 250°C and an equlibation time of 0.25 minutes.

Southern star	**S557.D**	**S558.D**	**S561.D**
Pressure (bar)	**150**	**200**	**250**
α-Humulene	36.82	40.31	34.23
β- Caryophyllene	16.31	14.73	11.59
Myrcene	9.03	15.06	32.13
Delta-Cadinene	6.58	4.94	2.62
α-Selinene	4.29	3.19	1.81
α-Amorphene	3.49	2.57	1.56
β-Selinene	3.38	2.53	1.44
α-Copaene	1.77	1.52	1.25
Southern star rerun	**S557.D**	**S558.D**	**S561.D**
Pressure (bar)	**150**	**200**	**250**
Alpha-Humulene	36.81	40.30	34.22
Beta-Caryophyllene	16.31	14.73	11.58
Myrcene	9.02	15.06	32.13
Delta-Cadinene	6.57	4.94	2.62
Alpha-Selinene	4.28	3.19	1.81
Alpha-Amorphene	3.49	2.57	1.56
Beta-Selinene	3.38	2.53	1.44
Alpha-Copaene	1.77	1.52	1.25

Southern passion	**S559.D**
Pressure (bar)	**200**
α-Humulene	34.83
β-Caryophyllene	15.26
α-Selinene	9.93
Myrcene	9.27
β-Selinene	7.86
delta-Cadinene	5.42
α-Amorphene	3.26
α-Copaene	1.51

African Queen	**S560.D**
Pressure (bar)	**200**
α-Humulene	34.68
Myrcene	21.96
β-Caryophyllene	15.32
α-Selinene	5.31
β-Selinene	4.26
Delta-cadinene	4.01
α-Amorphene	2.04
α-Copaene	1.49

Southern Promise	**S562.D**
Pressure (bar)	**150**
α-Humulene	30.05
β-Caryophyllene	16.30
Myrcene	12.95
α-Selinene	10.03
Selina-3,7(11)-diene	6.39
Delta-cadinene	5.07
α-Amorphene	3.32
α-Copaene	1.94