



NOVELBALTIC

Market driven authentic non-timber forest products from the Baltic Sea region

Non-Timber Forest products WP6 - Conceptual design of selected NTFP processes

O6.4. Engineering package for investment

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Elham Khalati
Pekka Oinas
Oliwer Sliczniuk

Abstract

In this report, the supercritical CO₂ extraction process models are shown for each type of raw material. In the following, the operation is described based on the process flow diagrams. These diagrams are developed according to the simulation results in Aspen Plus.

Process design and simulation play an important role in equipment selection and design. After equipment selection, the process equipment sizing is carried out based on relevant literature and vendor data or using design software including Aspen Exchanger Design and Rating. For process safety analysis, HAZOP study is conducted for the supercritical CO₂ extraction and product and solvent recovery steps. In this study, pressure, temperature, flow, and level are process parameters selected for each node.

For enhancing the health and safety of employees at the production plant, the industrial hygiene is studied from three aspects: engineering solutions, work practice controls, and administrative controls. Finally, sustainability and environmental concerns are analyzed for process waste and vent streams. The supercritical CO₂ extraction is a closed loop process, and most of CO₂ and co-solvents are recovered and reused in the extraction step. Therefore, there are not significant greenhouse gas (GHG) emissions.

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1. Engineering design

1.1. Extraction process simulation

For more detailed engineering design, the main extraction process has been modelled in Aspen Plus for each of the raw materials. For more accurate design, suitable property methods have been chosen based on the stream composition and operating pressure. Based on the simulation results, process flow diagrams are developed, and operating data for equipment and stream properties are used for equipment selection and sizing. Fig. 1 to 3 show the process simulation of the extraction process for roseroot, maral root, and garden angelica.

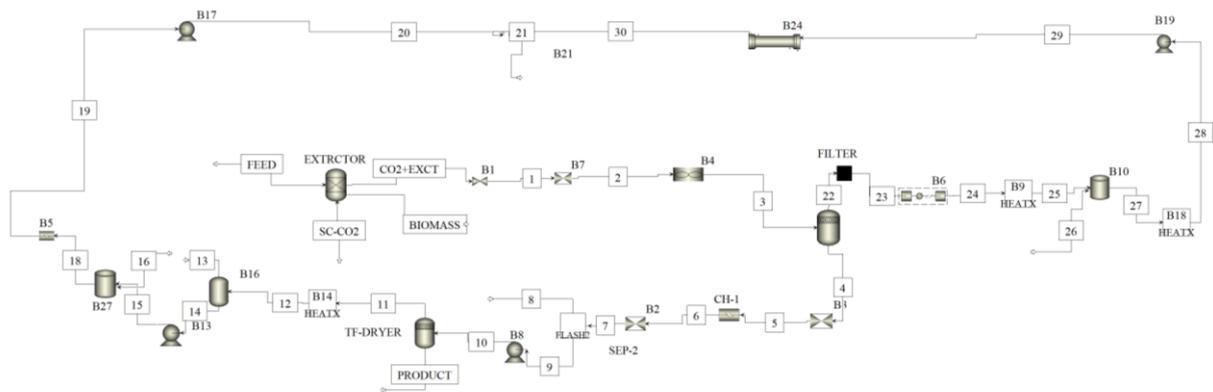


Fig. 1. Supercritical CO₂ extraction flowsheet for roseroot

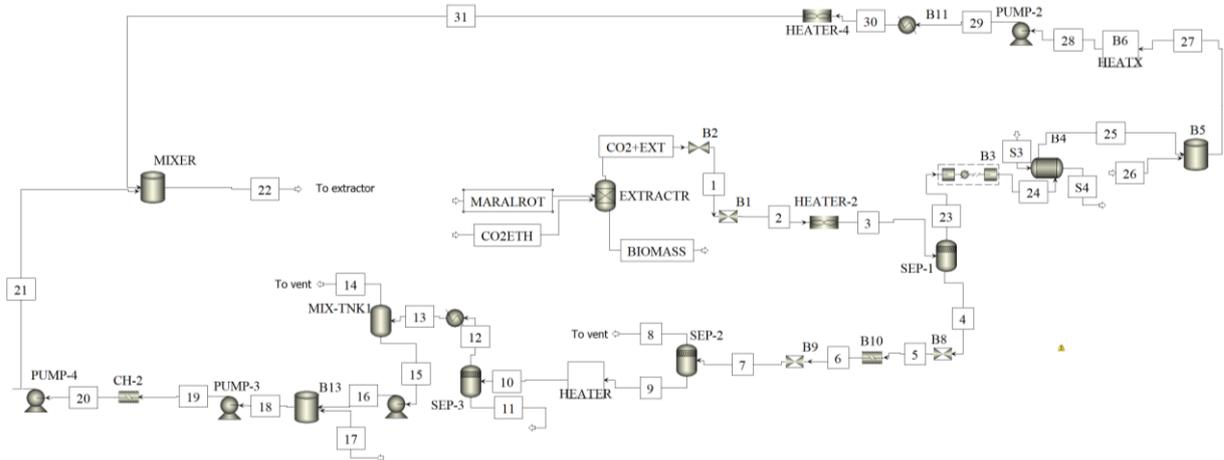


Fig. 2. Supercritical CO₂ extraction flowsheet for maral root

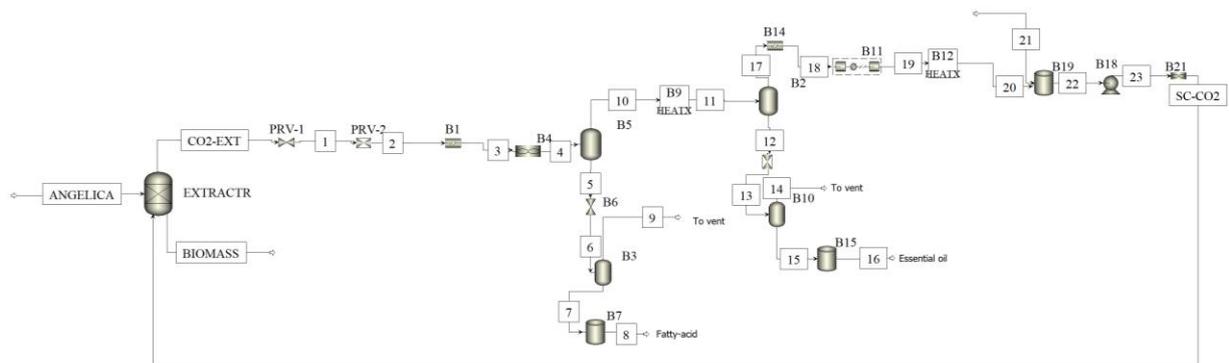


Fig. 3. Supercritical CO₂ extraction flowsheet for garden angelica

1.2. Process description

Developed process flow diagrams (PFDs) for pre-treatment of raw material, supercritical CO₂ (SC-CO₂) extraction, and solvent and product recovery are shown in appendices 1, 2, and 3. In the pre-treatment section, harvested plants are loaded in the silo FB-001, and they are transferred to the pre-washing section, WASH-001 and WASH-002. After initial cleaning, fresh herbs are cut into smaller pieces in KB-001. They enter the final washing step, WASH-003. Next, the plant cuts are dewatered in HD-001 by a vibrating screen. Clean herbs are stored in the intermediate silo, FB-002. Roseroot and maral root are needed to be dried before processing. Hence, they are moved from FB-002 to infrared radiation (IR) dryers, AA-001-A/F. Fresh cuts are dried for 3-5 h until the final moisture is ≤ 5 wt. %. Afterwards, the dried materials are bagged into 25 kg batches and stored in a container that is located in the OSBL area. In case of garden angelica, the fresh cuts are directly transferred to the extraction process plant from FB-002 without drying or storing them for a long time.

For SC-CO₂ extraction, recovered, liquified CO₂ is mixed with liquid and fresh CO₂, stored in FA-101 at 50 bar, in FA-102 at temperature 10°C. A cooling coil is considered inside FA-102 to keep the outlet stream temperature at 5°C. CO₂ is then pressurized with pump GA-101 to the desired extraction pressure, depending on the type of raw material. An internal cooling system has been considered in GA-101 to prevent CO₂ from vaporization during pressurizing. In the following, pressurized CO₂ is heated in EA-101 to reach the extraction temperature. In case of roseroot and maral root, co-solvent is also used to increase the extraction efficiency. The recovered co-solvent is mixed with fresh co-solvent in liquid state in FA-104 at 1 bar. Co-solvent is then pressurized with pump GA-102 to reach CO₂ pressure. In the following, pressurized CO₂ and co-solvent are divided into two streams and the divided streams are mixed in two static mixers GD-101-A/B. Two parallel SC-CO₂ and co-solvent mixture streams enter the extraction columns DB-101-A/B and contact the loaded raw material. The total extraction process is 3h, and during the operation SC-CO₂ (in some cases with co-solvent) flows through the column continuously. Temperature and pressure of the extraction process are 80 °C and 200 bar for roseroot, 60 °C and 280 bar for maral root, and 40 °C and 120 bar for garden angelica.

After the extraction vessels, DB-101-A/B, the outlet stream, including CO₂, extract, and also in some cases co-solvent, is depressurized in two stages and CO₂ enters the subcritical region. In the second pressure reducing valve, an electrical heating element is considered around the valve to prevent it from freezing. After depressurizing, depending on the type of plant, the order of separation is different. In case of garden angelica, the pressure of the main stream reduces to 10 bar, and then its temperature is increased in the heat exchanger EA-102. Next, fatty acids, heavier materials, are separated from the main stream in cyclone separator HD-101 and are collected in FA-107 at 1 bar. The outlet gas stream of HD-101 is cooled in heat exchanger EA-103. In separator HD-102, essential oil, the main product, is separated from CO₂ gas. Essential oil is collected in vessel FA-109 at 1 bar.

In case of roseroot and maral root, after depressurization to 35 bar, temperature is increased to 35 °C in EA-102. The main stream does not enter HD-101, and it goes through the stream line S23. Gaseous CO₂ is separated from co-solvent and extract in HD-102. Co-solvent and extract mixture is depressurized to 1 bar while keeping the temperature at 25 °C with an electrical heating element. To separate co-solvent from the extract, the mixture flows into vertical thin film dryers AA-101-A/B. Then the main product, in the solid phase, is collected in product collectors FB-101-A/B which are located at bottom of AA-101-A/B. Thin film dryers operate at vacuum pressure, and it is controlled by vacuum pump GC-101. The top product of AA-101-A/B is almost pure co-solvent where it is condensed and recycled back to mix with fresh co-solvent in FA-104.

In all cases, the top product of HD-102 is gaseous CO₂. CO₂ is compressed to 50 bar in GB-101. Pressurized CO₂ is condensed in EA-104-A/B. Then it is cooled down to 10 °C. In the following, recovered CO₂ is mixed with fresh CO₂ in vessel FA-102.

2. Equipment type and dimension

Table 1 shows the type and dimensions of the main equipment used in pre-treatment and the main production process. Equipment is selected and sized based on literature review and vendor quotes or using software such as Aspen Exchanger Design and Rating (Aspen EDR). [1–3] Based on the chemical compatibility chart for available components in the process, stainless steel-304 is chosen as the main material. In some cases, based on the manufacturer specifications, equipment material is stainless steel-316. Based on the equipment information, capital investment cost is estimated.

Table 1. Equipment information and dimensions

Tag no.	Equipment	Quantity	Heating area (m ²)	Volume (m ³)	W (m)	L (m)	H (m)	Material
Pre-treatment section								
FB-001	Hopper	1		2	1.5		1.7	SS-304
JF-001	Screw feeder	1			0.5	1.6		SS-304
WSH-001	Drum vegetable washing machine	1			0.95	1.5	1.14	SS-304
WSH-002	Vegetable washing machine	1			1.2	5	1.3	SS-304
KB-001	Root cutter	1			0.9	1.9	1.25	SS-304
WSH-003	Vegetable washing machine	1			1.2	2.5	0.6	SS-304
HD-001	Vibration dewatering machine	1			1.2	2.5	0.6	SS-304
FB-002	Hopper	1		2	1.5		1.7	SS-304
JF-002	Vibrating feeder	1			0.5	1.6		SS-304
AA-001-A/F	Infrared dryer	6	25		0.75	1.6	2.3	SS-304
Extraction process								
FA-101	CO2 cyliner	37			0.23		1.46	-
FA-102	Vertical vessel	1		0.26	0.41		2	SS-304
GA-101	Diaphragm pump	1	-	-	1.07	1.19	1.21	SS-316
-	Mass flow meter	1			0.14	0.21	0.27	SS-316
EA-101	SM electrical heater	10	0.1		0.02	1.7		SS-304
FA-103	drum	1		0.25	0.58		1.05	HDPE
FA-104	Vertical vessel	1		0.022	0.24		0.5	SS-304
GA-102	Plunger pump	1	-	-	0.6	0.95	0.9	SS-316
KA-101	Grinder	1			0.6	0.75	1.45	SS-304
DB-101-A/B	Extractor	2	-	0.33	0.4	-	2.2	SS-304
EA-102-A/D	SM electrical heater	4	0.1		0.02	1.2	-	SS-304
HD-101	Cyclone separator	1	-		0.1	-	0.4	SS-304
HD-101	Vertical vessel	1		0.001	0.1		0.15	SS-304
FA-107	Vertical storage vessel	1		0.003	0.12		0.24	SS-304
EA-103	Plate heat exchanger	1	0.20		0.13	0.03	0.43	SS-304
HD-102	Cyclone separator	1	-	-	0.2		0.8	SS-304
FA-108	Static mixer electrical heater	1		0.001	0.1		0.15	SS-304
FA-109	Vertical vessel	1		0.005	0.15	-	0.3	SS-304
HA-101	Compressed gas filter	1			0.05		0.1	SS-304
GB-101	Two stage compressor	1			0.7	1.42	1.06	SS
EA-104-A/B	Plate heat exchanger	2	0.9	-	0.13	0.11	0.43	SS-304
FA-110	Separator	1		0.01	0.15		0.5	SS-304
GA-103	Gear pump	1			0.17	0.41	0.23	SS-304
AA-101-A/B	Thin film dryer	2	1.2		0.29		1.5	SS-304
FB-101-A/B	Bin	2			0.38		0.35	SS-304
EA-105	Plate heat exchanger	1	0.8		0.27	0.06	0.57	SS-304
FA-111	Vertical vessel	1		0.022	0.24		0.49	SS-304
GC-101	Vacuum pump	1			0.17	0.45	0.26	SS-304
GA-104	Gear pump	1			0.17	0.41	0.23	SS-304

3. Process safety analysis

As a process safety analysis, HAZOP study is conducted for the process parameters related to the SC-CO₂ extraction and solvent and product recovery sections based on the developed piping and instrumentation (PI) diagrams. PI diagrams are shown in Appendices 4, 5, and 6. In this study, pressure, temperature, flow rate, and level are main parameters considered for HAZOP study. HAZOP study for SC-CO₂ extraction section and product and solvent recovery section can be provided as separate documents.

4. Industrial hygiene

Industrial hygiene includes identifying, assessing, and dealing with workplace hazards to prevent employees from injury and illness. [4] Engineering solutions, workplace analysis and monitoring, and administrative controls are the main means of minimizing occupational hazards. [4,5]

Engineering solutions minimize employee exposure by mitigating or eliminating the hazard at the source or isolating the operator from the hazard. In the SC-CO₂ extraction process, based on the HAZOP study, safety control and alarm systems have been considered to prevent from parameter deviations and minimize any operating risks that can threaten employees. In case of potential hazards related to chemicals, above a specific level of CO₂ higher than permissible exposure limit for daily workplace exposures, it can be harmful due to a toxic atmosphere or oxygen deprivation. Therefore, installation of ventilation systems, toxic and O₂ depletion detectors, and alarms are necessary.

Work practice controls define the employee manner during the work. Some fundamental and easily implemented work practice controls include

- replacing the common work routines with suitable procedures that reduce the occupational hazards during the production time or control of the equipment.
- Inspecting and maintaining the process equipment, control systems, and safety instruments regularly
- Prohibiting improper habits such as eating, drinking, and smoking in regulated areas

Administrative controls include reducing the exposure levels by scheduling production and tasks of the employers properly.

5. Environmental concerns

In the pre-treatment section, wastewater of the washing section includes removed dirt and debris from the harvested plants; therefore, it does not include any dangerous material. In case of combining IR drying with air convection, the wet air includes removed moisture and partially dried plant's natural components which are not harmful. However, in some cases, if the plant is aromatic, outlet air smell can be strong, and an odor control system can be installed at the air exhaust.

In contrast to conventional methods such as solvent extraction, SC-CO₂ extraction process requires little or in some cases no organic solvent. Therefore, it avoids the organic solvent waste incineration or any similar action that is an environmentally hazardous and expensive process. [6]

In addition, the main production plant is a closed process, and most of CO₂ and co-solvents are recovered and recycled within the process for reuse in the extractor. Therefore, the discharged CO₂ and co-solvents

through the ventilation system are negligible, and there are not significant greenhouse gas (GHG) emissions to the atmosphere. In addition, other gaseous materials in the vent stream are part of the utilized herbs, and they naturally exist in the environment. Hence, they do not have negative effects. For further considerations, an odor control system can be installed in the venting line.

The remaining biomass waste is not in contact with hazardous chemicals during the operation. Therefore, it can directly be transferred to the storage area and be used for other applications.

6. Conclusion

Based on the process design of SC-CO₂ extraction in Aspen Plus, operational data is available for equipment selection and design. This information provides a basis for capital and operational costs assessment. From the sustainability aspect, SC-CO₂ extraction is an eco-friendly process. For further considerations, installing odor control systems are preferred at the air exhaust of IR dryer and ventilation line of the main production process. For process safety analysis and employee safety, HAZOP study is conducted, and three main aspects of industrial hygiene are investigated.

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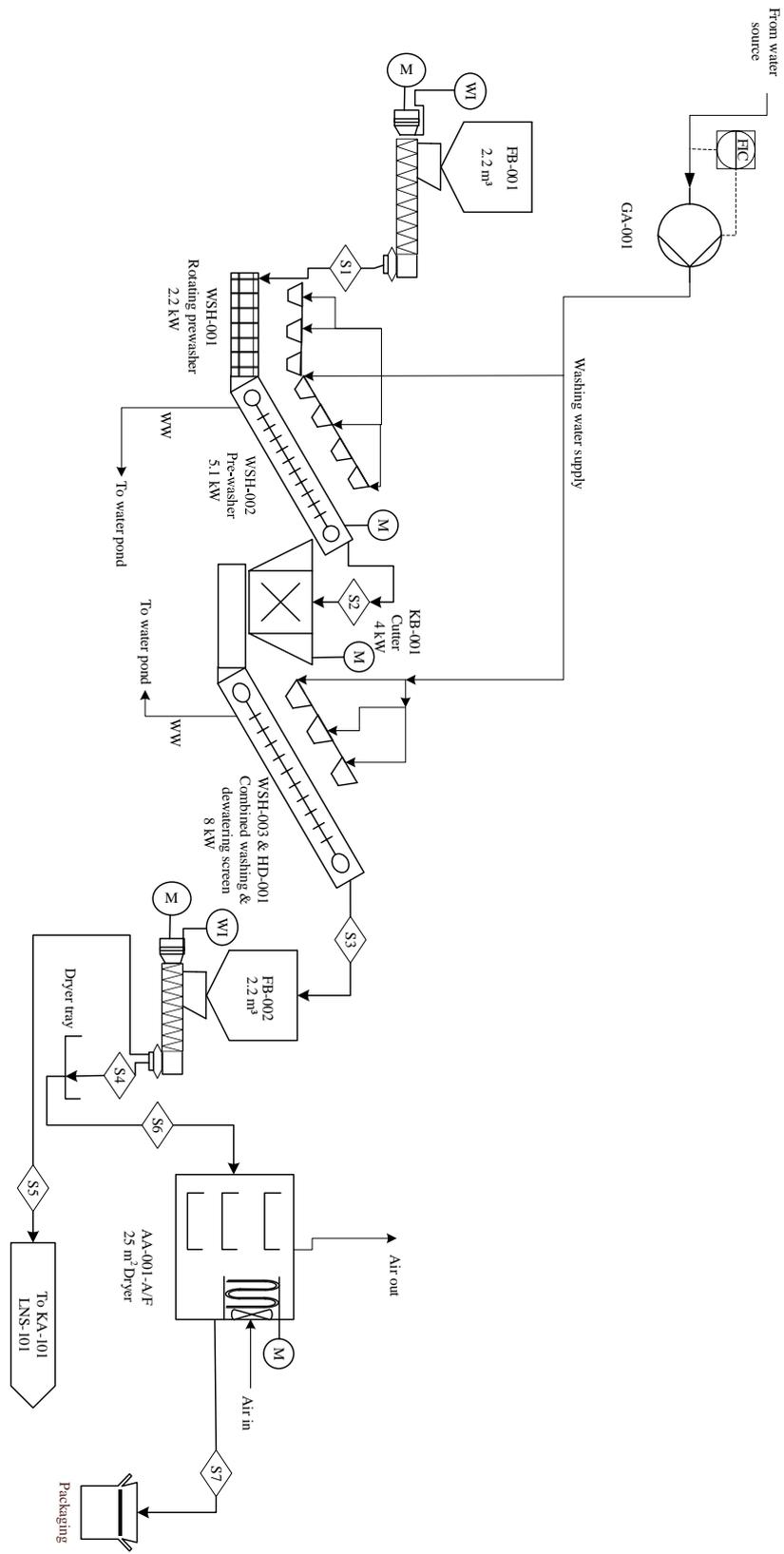
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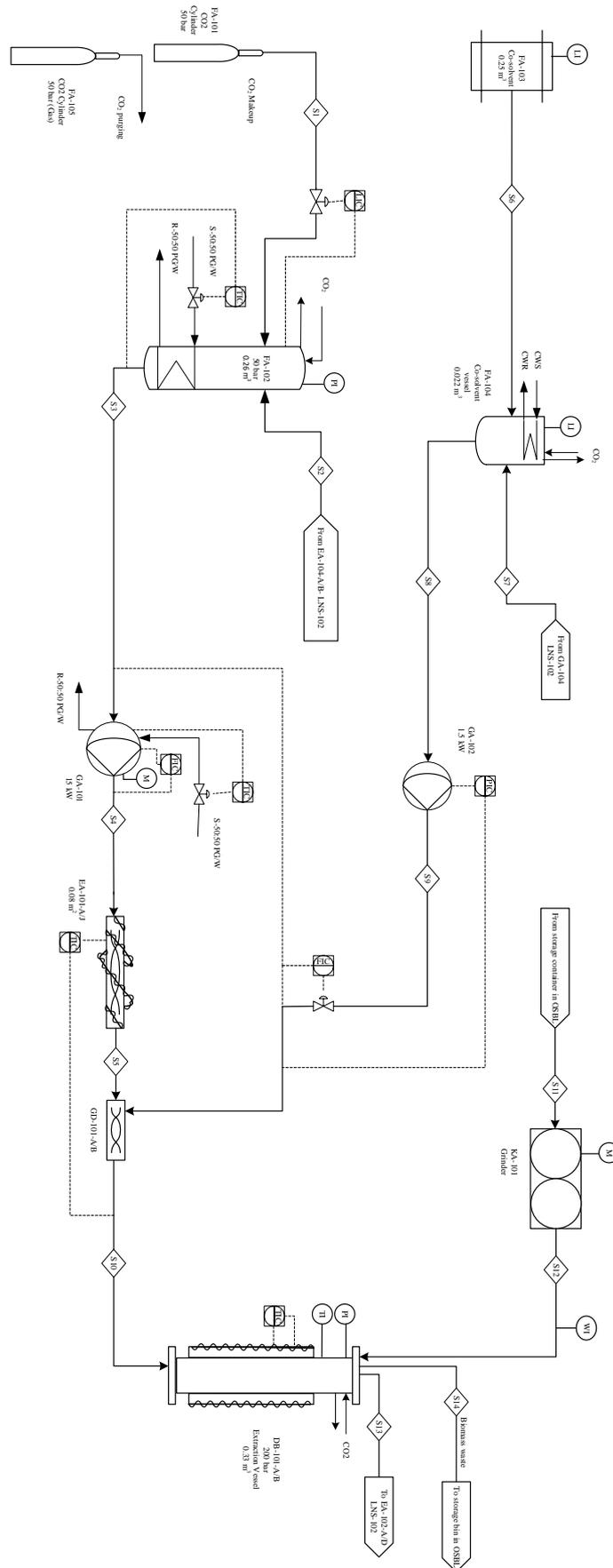
Appendices



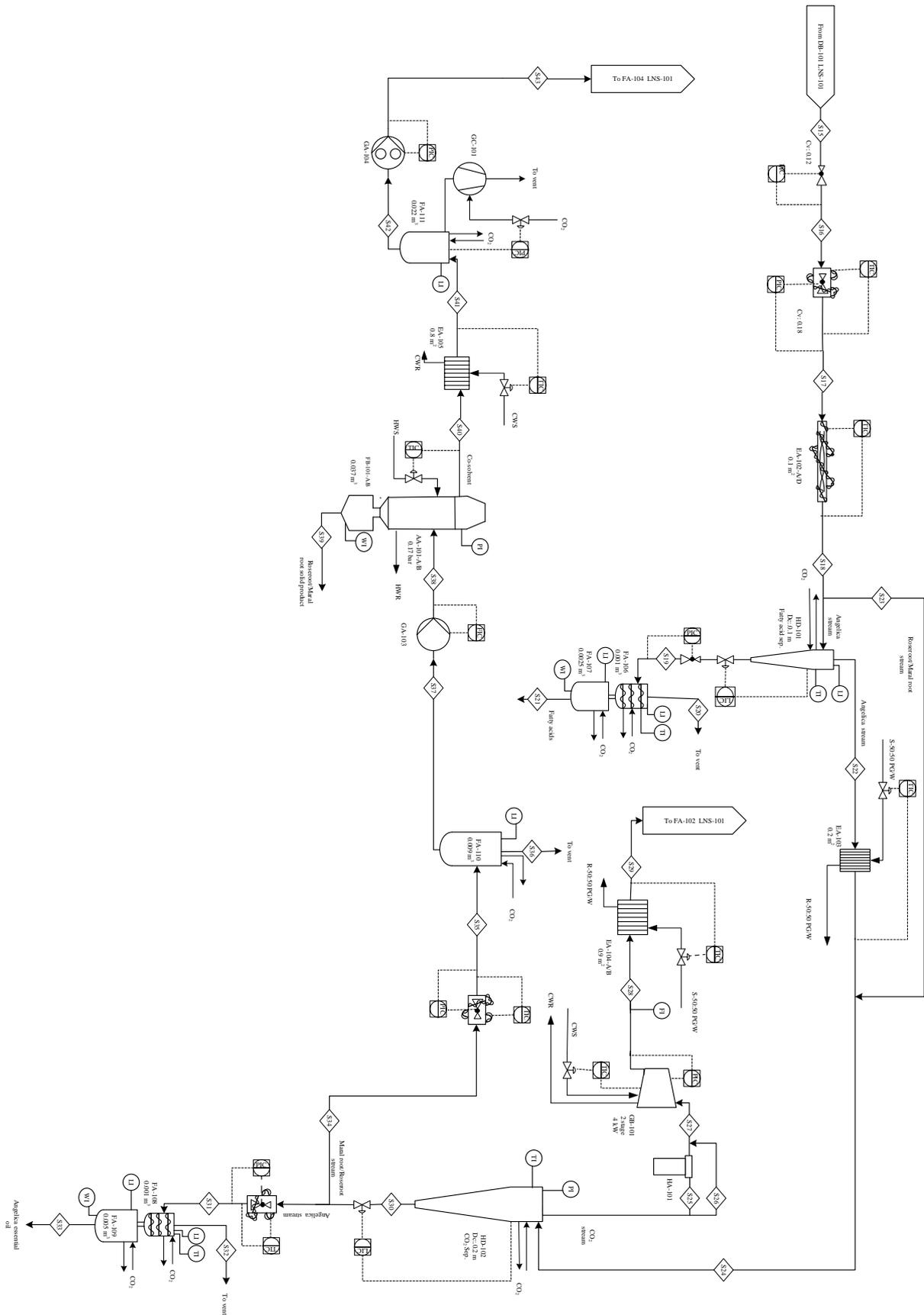
Appendix 1. Process flow diagram of the pre-treatment section



Appendix 2. Process flow diagram of the SC-CO₂ extraction section



Appendix 3. Process flow diagram of the product and solvent recovery section



Appendix 6. PI diagram of co-solvent and extract recovery in vacuum conditions

