NOVELBALTIC

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

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

O6.3. Process flow diagrams (PFD)

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Abstract

In this report, the overall configuration of the process is introduced. The extraction process consists of two main steps: pre-treatment and the main extraction process. In this process, annually three different products are extracted from three different plants, roseroot, maral root, and garden angelica. The total production capacity of the process plant is approximately 13 tons.

Comprehensive process flow diagrams are prepared for pre-treatment, supercritical CO_2 extraction, and product and solvent recovery steps. The diagrams include process piping, main equipment, related control systems, outside-battery limit connections, and CO_2 and co-solvent recycle streams. In addition, stream tables in process flow diagrams present the mass balance and operating information.

For safety reasons, the main production process is located inside an ATEX certified container with 40 m^2 area. Based on the plant layout, the total process plant area including outside and inside battery limits is 600 m^2.

Contents

1. Definition of the selected process

Extraction of intended products from raw materials includes two general steps: pre-treatment of the harvested herbs and the main extraction process. The pre-treatment step includes prewashing, cutting, washing, dewatering, and in case of roseroot and maral root, they are dried after the dewatering stage. The drying method chosen for this process is infrared radiation (IR), with the possibility of combining with hot air convection. According to Pääkkönen et al. (1998), infrared radiation is a potential method for drying herbs since it operates at low drying temperature and shortens the processing time. [1] The dried materials are bagged in 25 kg bags and stored in an area with favorable conditions. Pre-treated fresh herbs, garden angelica, are directly transferred to the main extraction process plant, and their storage time is negligible in comparison to the dried herbs. Dried and fresh raw materials are ground with a continuous grinder before loading them in the extractors.

The extraction columns are two high-pressure vessels. The solid materials are placed inside the extractors from the top of the column. The extraction process is semi-continuous and supercritical CO_2 stream flows continuously through the extractor and contacts solid particles. Metal meshes are considered at the top and bottom of the column to keep the solid particles in the extractor during the operation. After the extraction, the biomass waste is removed from the top of the extractor and is transferred to the storage bin. The leftover biomass can be used as a heat source or raw material for other processes including ethanol fermentation and producing bio-oil. [2]

In this process, electrical elements are wrapped around the heat exchangers to heat the process streams in different parts of the process. The cooling is done with cooling water when the outlet temperate of the hot stream is above 30°C, for lower temperatures propylene glycol -water (50:50) mixture is utilized. Cooling water utility system and Refrigeration unit are considered in the OSBL section to provide the required utility.

Based on the literature review, after the extraction step, the different components are separated from each other by controlling the temperature and the pressure in separators. In case of roseroot and maral root, to recover co-solvent and dry the final product, two thin film dryers are considered in the last step of the product recovery unit. This type of dryer has a cylindrical, vertically arranged body with a heating jacket and a rotor inside. [3–5]

2. Capacity and production

The extraction capacity of the process plant is processing 200 kg raw material per batch, and a daily capacity of the process plant is 600 kg, based on 3 batches per day. Table 1 shows the annual raw material consumption and production capacity of the plant. Based on the literature review, extraction yields for roseroot, maral root, and garden angelica are 18.4 wt. %, 1.1 wt. %, and 0.2 wt. % based on the total loaded raw material into the extractor. [3–5] The total annual production capacity of the plant is approximately 13 tons from three plants. [6,7]

Table 1. Annual production capacity of the plant

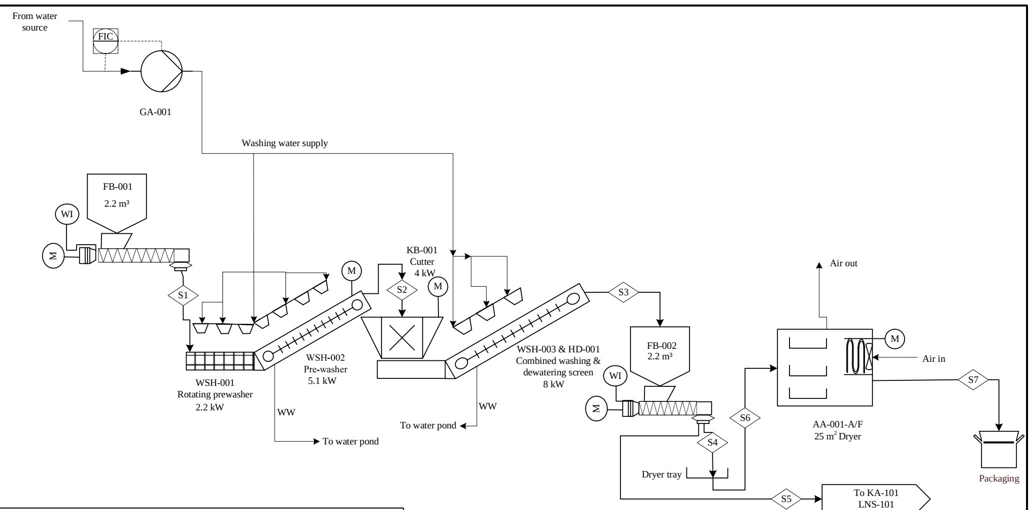
Herb	Raw material consumption	Production
	(t/a)	(t/a)
Roseroot (dried)	69.3	12.75
maral root (dried)	37.8	0.42
Garden angelica (fresh)	37.8	0.076
Total	144.9	13.246

3. Process description

The process is divided into three sections. three sections are pre-treatment, supercritical CO_2 extraction, product and solvent recovery.

3.1. Pre-treatment of raw material

Fig. 1 shows the process flow diagram (PFD) of the pre-treatment section. This PFD includes process main pipelines, major equipment equipped with main control systems, and the related stream table presenting operational data. The harvested plants are loaded in the silo FB-001, and they are transferred to the pre-washing section, WASH-001 and WASH-002. After the initial cleaning, fresh herbs are cut into smaller pieces in KB-001, and they enter the final washing step, WASH-003. Next, the washed material is 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 IR dryers, AA-001-A/F, where herb cuts are dried for 3-5 h until the water percentage of the mass is ≤ 5%. 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.



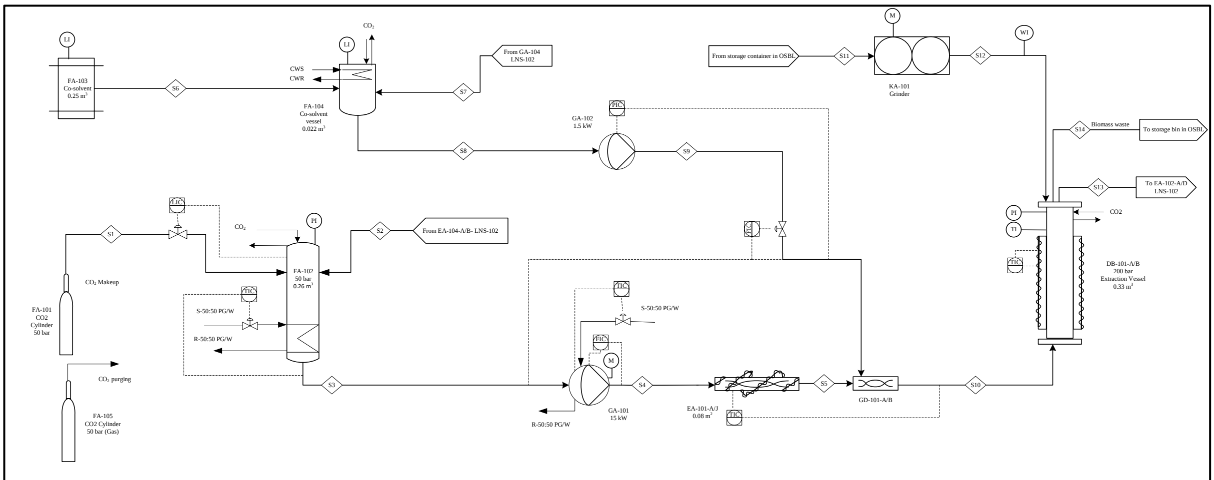
Parameters	Streams						
	S1	S2	S3	S4	S5	S6	S7
	Roseroot						
Total mass (kg)	864	864	864	864		864	200
Biomass (kg)	148	148	148	148		148	148
Extract (kg)	42.3	42.3	42.3	42.3		42.3	42.3
Water (kg)	674	674	674	674		674	10
Fatty acid (kg)							
Density (kg/m3)	830	830	830	830		830	626
Pressure	1	1	1	1		1	1
Temperature (°C)	20	20	20	20		20	24
	Maral root						
Total mass (kg)	760	760	760	760		760	200
Biomass (kg)	179.5	179.5	179.5	179.5		179.5	179.5
Extract (kg)	10.56	10.56	10.56	10.56		10.56	10.56
Water (kg)	570	570	570	570		570	10
Fatty acid (kg)							
Density (kg/m3)	800	800	800	800		800	600
Pressure	1	1	1	1		1	1
Temperature (°C)	20	20	20	20		20	24
	Angelica root						
Total mass (kg)	200	200	200	200	200		
Biomass (kg)	49	49	49	49	49		
Extract (kg)	0.7	0.7	0.7	0.7	0.7		
Water (kg)	150	150	150	150	150		
Fatty acid (kg)	0.25	0.25	0.25	0.25	0.25		
Density (kg/m3)	800	800	800	800	800		
Pressure (bar)	1	1	1	1	1		
Temperature (°C)	20	20	20	20	20		

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

3.2. Supercritical extraction

Fig. 2 shows the process flow diagram of the supercritical CO_2 extraction of valuable products. This process is a close loop process. Recovered, liquified CO_2 is mixed with liquid and fresh CO_2, stored in FA-101 at 50 bar, in vessel FA-102 at temperature 10°C. A cooling coil is considered inside FA-102 to keep the outlet stream temperature at 5°C. The fresh and recycled CO2 mixture 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 CO_2 from vaporization during pressurizing. In the following, pressurized CO_2 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, and the co-solvent mixture is pressurized with pump GA-102 to reach CO_2 pressure. In the following, pressurized CO_2 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-CO_2 and co-solvent mixture streams enter the extraction columns DB-101-A/B and contact the loaded raw material. The extraction process is a semi-continuous process, and the column is manually filled with plant mass by opening the top of the column and putting a 200 kg batch in it before starting the process. The total extraction process is 3h, and during the operation SC-CO_2 (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 operation, the biomass waste is removed from the top of the column and the column is prepared for the next extraction batch.



Parameters	Stream number													
	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11	S12	S13	S14
	Roseroot													
Total mass (kg)	1	1895	1896.0	1896.0	1896.0	4	186	190	190	2086	200	200	2123	162
CO2 (kg)	1	1895	1896	1896	1896					1896			1896	
Ethanol (kg)														
Biomass (kg)											148	148		148
Extract (kg)											42	42	37.8	5
Water (kg)						4	186	190	190	190	10	10	190	10
Fatty acid (kg)														
Density (kg/m3)	864	743	789	744	516	994	980	980	722	555	626	626	561	626
Pressure	50	50	50	200	200	1	1	1	200	200	200	200	200	200
Temperature (°C)	10	10	5	41	81	25	40	40	88	80	80	80	80	80
	Maral Root													
Total mass (kg)	14	309	323	323	323	1.5	19.5	21	21	344	200	200	346	198
CO2 (kg)	14	307	321	321	321					321			321	
Ethanol (kg)		1.8	1.8	1.8	1.8	1.5	19.5	21	21	23			23	
Biomass (kg)											179.46	179.46		179.5
Extract (kg)											10.56	10.56	2.2	8.4
Water (kg)											10	10		10
Fatty acid (kg)														
Density (kg/m3)	864	752	795	783	752	799	794	795	631	771	600	600	792	600
Pressure	50	50	50	280	280	1	1	1	280	280	280	280	280	280
Temperature (°C)	10	10	5	50	57	25	29	29	78	60	60	60	60	60
	Angelica root													
Total mass (kg)	0.15	321	321	321	321					321	200	200	322	199
CO2 (kg)	0.15	321	321	321	321					321			321	
Ethanol (kg)														
Biomass (kg)											49	49		49
Extract (kg)											0.7	0.7	0.43	0.27
Water (kg)											150	150		150
Fatty acid (kg)											0.25	0.25	0.14	0.11
Density (kg/m3)	864	864	897	734	607					607	800	800	607	800
Pressure (bar)	50	50	50	120	120					120	120	120	120	120
Temperature (°C)	10	10	5	25	40					40	40	40	40	40

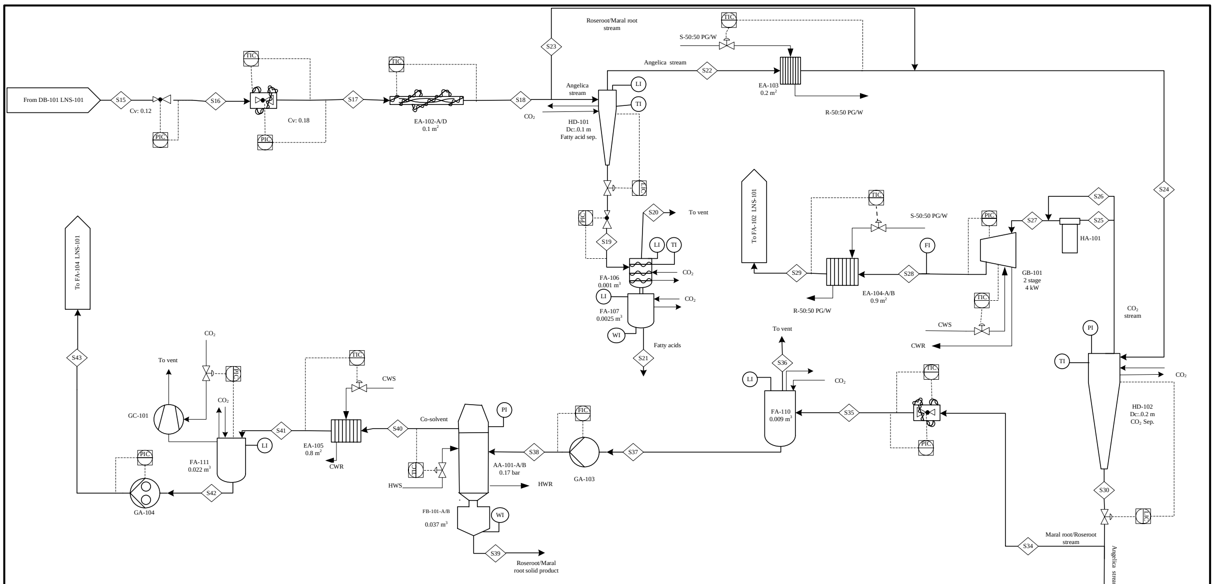
Fig. 2. Process flow diagram of the supercritical CO_2 extraction

3.3. Product and solvent recovery

Fig. 3 shows the process flow diagram of the product and solvent recovery process. After the extraction vessels, DB-101-A/B, the outlet stream, including CO_2, extract, and also in some cases co-solvent, is depressurized in two stages and CO_2 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 after 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-101is cooled in heat exchanger EA-103, and essential oil, the main product, is separated from CO_2 gas in separator HD-102 and is collected in vessel FA-109 at 1 bar.

In case of roseroot and maral root, after depressurization, the pressure is decreased to 35 bar and temperature is increased to 35 °C in EA-102. In the following, CO_2 gas 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 element wrapped around the pressure reducing valve. To separate co-solvent from the extract, the mixture flows into vertical thin film dryers AA-101-A/B, and 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 CO_2, which is first compressed to 50 bar in GB-101 and condensed in EA-104-A/B and cooled down to 10 °C. In the following, recovered CO_2 is mixed with fresh CO_2 in vessel FA-102. In case of roseroot, an inline adsorber has been considered to remove the remained water from CO_2.



Parameters	S15	S16	S17	S18	S19	S20	S21	S22	S23	S24	S25	S26	S27	S28	S29
Roseroot															
Total mass (kg)	2123	2123	2123	2123					2123	2123	1897		1895	1895	1895
CO2 (kg)	1896	1896	1896	1896					1896	1896	1895		1895	1895	1895
Ethanol (kg)															
Biomass (kg)															
Extract (kg)	38	37.8	37.8	37.8					37.8	37.8					
Water (kg)	190	190	190	190					190	190	1.6				
Fatty acid (kg)															
Density (kg/m3)	561	359	89	81					81	81	73		73	112	743
Pressure	200	100	35	35					35	35	35		35	50	50
Temperature (°C)	80	53	20	35					35	35	35		35	40	10
Maral root															
Total mass (kg)	346	346	346	346					346	346		309	309	309	309
CO2 (kg)	321	321	321	321					321	321		307	307	307	307
Ethanol (kg)	23	23	23	23					23	23		2	2	2	2
Biomass (kg)															
Extract (kg)	2.2	2.2	2.2	2.2					2.2	2.2					
Water (kg)															
Fatty acid (kg)															
Density (kg/m3)	792	712	93	81					81	81		73	73	113	752
Pressure	280	150	35	35					35	35		35	35	50	50
Temperature (°C)	60	48	20	35					35	35		35	35	40	10
Angelica root															
Total mass (kg)	322	322	322	322	0.2	0.03	0.16	321.4		321.4		320.9	320.9	320.9	320.9
CO2 (kg)	321	321	321	321	0.03	0.03		321		321		320.9	320.9	320.9	320.9
Ethanol (kg)															
Biomass (kg)															
Extract (kg)	0.43	0.43	0.43	0.43	0.02		0.02	0.4		0.4					
Water (kg)															
Fatty acid (kg)	0.14	0.14	0.14	0.14	0.14		0.14								
Density (kg/m3)	607	435	19	17	11	2	836	16.50		20		20	20	123	739
Pressure (bar)	120	60	10	10	1	1	1	10		10		10	10	50	50
Temperature (°C)	40	22	20	60	44	44	44	60		5		5	5	30	10

Parameters	S30	S31	S32	S33	S34	S35	S36	S37	S38	S39	S40	S41	S42	S43
Roseroot														
Total mass (kg)	227				227	227	1	226	226	40	186.2	186.2	185.9	186
CO2 (kg)	1				1	1	1	0.3	0.3		0.3	0.3		
Ethanol (kg)														
Biomass (kg)														
Extract (kg)	37.8				38	38		38	38	38				
Water (kg)	188				188	188		188	188	2.1	185.9	185.9	185.9	185.9
Fatty acid (kg)														
Density (kg/m3)	812				812	1040	1.8	1040	1040	1321	0.1	566	980	980
Pressure	35				35	1	1	1	1	0.17	0.17	0.17	0.17	1
Temperature (°C)	35				35	25	25	25	25	57	57	40	40	40.1
Maral root														
Total mass (kg)	37				37	37	14.8	22.1	22.1	2.3	20	19.8	19.7	19.7
CO2 (kg)	14				14	14	14	0.1	0.1		0.1	0.1		
Ethanol (kg)	21				21	21	1.2	20	20	0.12	19.7	19.7	19.7	19.7
Biomass (kg)														
Extract (kg)	2.2				2.2	2.2		2.2	2.2	2.2				
Water (kg)														
Fatty acid (kg)														
Density (kg/m3)	762				762	4	1.8	832	832	1258	0.6	93.1	794	794
Pressure	35				35	1	1	1	1	0.35	0.35	0.35	0.35	1
Temperature (°C)	35				35	25	25	25	25	53	53	29	29	29
Angelica root														
Total mass (kg)	0.5	0.5	0.1	0.4										
CO2 (kg)	0.1	0.1	0.1											
Ethanol (kg)														
Biomass (kg)														
Extract (kg)	0.4	0.4		0.4										
Water (kg)														
Fatty acid (kg)														
Density (kg/m3)	891	7.56	2	846										
Pressure (bar)	10	1	1	1										
Temperature (°C)	5	25	25	25										

Fig. 3. Process flow diagram of product and solvent recovery process

4. Plant layout

The layout of the plant includes production spaces for the whole process, storage areas, and waste collecting areas. The layout is divided into two different parts: Inside Battery Limits (ISBL) and Outside Battery Limits (OSBL). The functions differ in that ISBL focuses on the production spaces and what they need to operate, and OSBL shows the whole picture including ISBL and all other functions not directly attached to the production.

4.1. ISBL layout

ISBL is divided into pre-treatment and production sections. Fig. 4 shows the ISBL layout including the pre-treatment section and the production plant that is located inside an ATEX-certified container to make it portable and protect it from possible risks. The required area for ISBL is approximately 380 m^2. Fig. 5 and Fig. 6 show 3D and 2D layouts of the production plant. In the container, all the equipment is connected by piping, and enough spacing has been considered for maintenance and further inspections. CO_2 cylinder pallets are located outside of the container so that bottles can be refilled easily when necessary. The required area for the production container is 40 m^2, and its internal height is 2.6 m.

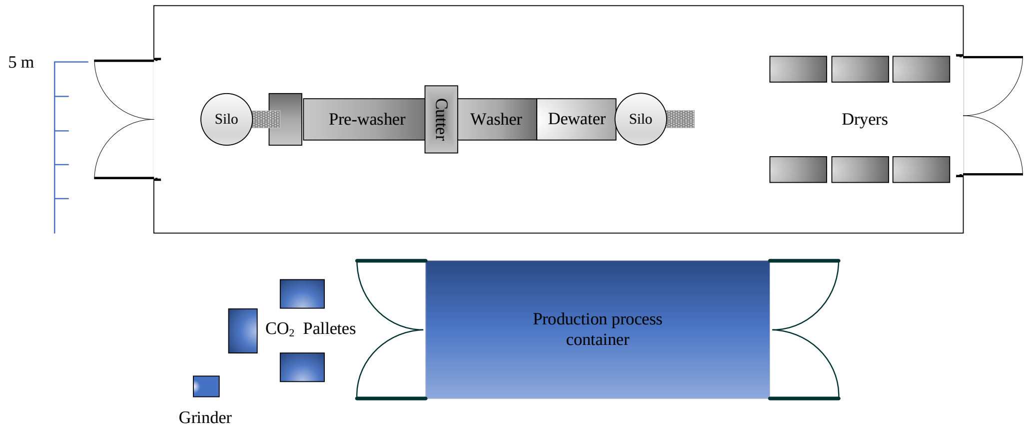


Fig. 4. ISBL layout

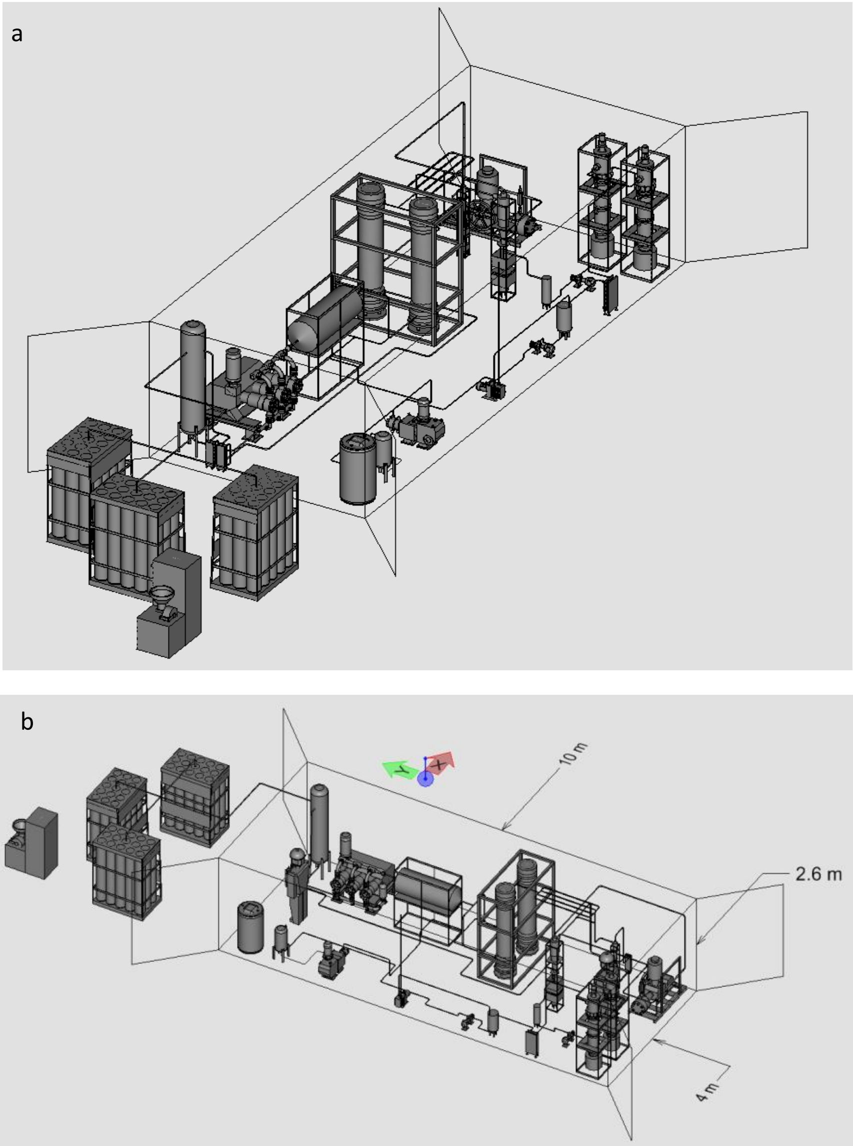


Fig. 5. 3D plant layout for the production plant a. Front view, b. side view

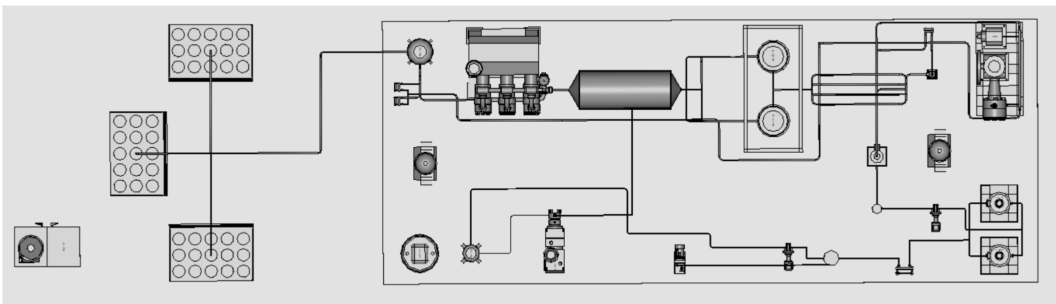


Fig. 6. 2D layout for the production plant

4.2. OSBL layout

Outside battery limit (OSBL) covers storage area for fresh plant stock, dried stock, products, and biomass waste. Utility units and wastewater collecting area are also presented. Fig. 7 shows the OSBL layout from the top view. After drying roseroot and maral root cuts, they are stored in a container with appropriate conditions to keep their quality during the year. A cold storage area is also considered for storing garden angelica for maximum 2 weeks. Storing conditions of dried and fresh material are different so they are not stored in a common area. Biomass waste is stored indoors to keep it from undesired conditions such as raining and snowing. It can be used for other application such as burning as fuel, ethanol fermentation, and producing bio-oil. A pond has been considered for collecting wastewater utilized for washing the harvested material. Since hazardous material does not exist in the wastewater, it can also be directed to a nearby lake or river. The required area for the whole plant is estimated to be 600 m^2.

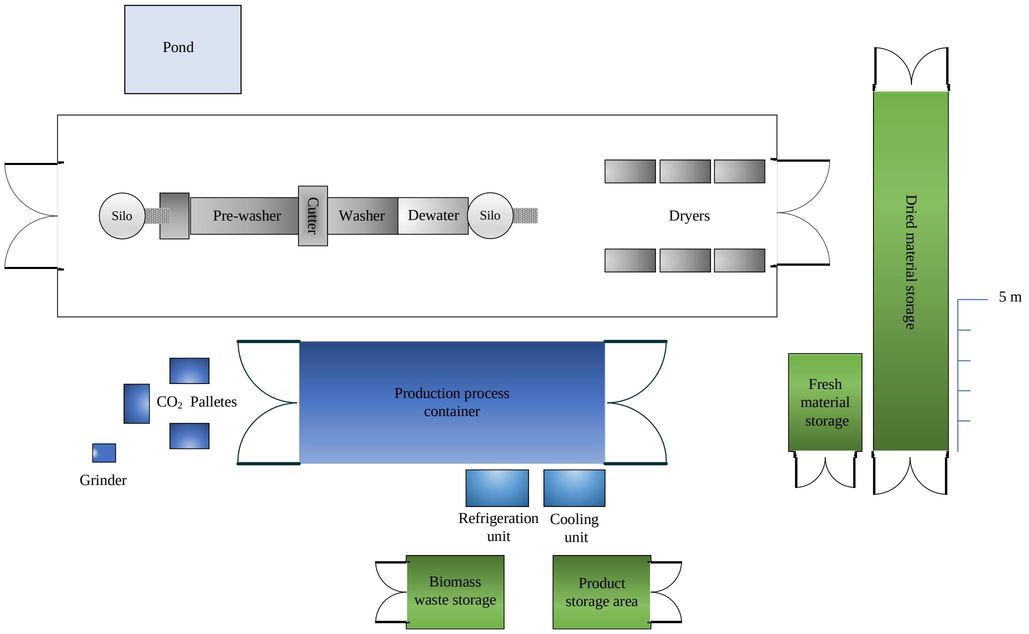


Fig. 7. OSBL layout

5. Conclusion

The proposed plant has an annual production capacity of approximately 13 tons. roseroot, maral root and garden angelica are three raw materials utilized in this process, and their annual consumption is estimated based on their availability, harvesting time, and storing conditions. The main products of the plant are essential oils from garden angelica and solid extracts from roseroot and maral root. Raw materials are directly transferred from fields to the plant, and they are pre-treated before the main extraction process.

Three extraction batches can be done daily. Each batch takes around 3 hours. Operating conditions of the process depend on the type of raw material. 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.

The process flow diagram is divided in three areas: pre-treatment of raw material, supercritical CO2 extraction, and product and solvent recovery. This diagram includes the main equipment and related control systems, process and utility pipelines, and stream tables including the operational information. For safety reasons, the main production plant is located in an ATEX certified container. Based on the plant layout, the total required area for the plant including the ISBL and OSBL is 600 m^2.

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