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Supercritical Extraction of Essential Oils of *Cymbopogon martini*, *Cymbopogon winterianus*, and *Eucalyptus citriodora* with Carbon Dioxide

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The extraction of essential oils of *Eucalyptus citriodora*, *Cymbopogon winterianus* and *Cymbopogon martini* has been studied using supercritical carbon dioxide as solvent. The raw materials were dried at room temperature ($30 \pm 2^\circ\text{C}$) for 5 days. The dried leaves were cut in pieces of 1cm (*Eucalyptus citriodora*) or 3cm of length (*Cymbopogon winterianus* and *Cymbopogon martini*). The fixed bed was packed with $87 \pm 1\text{g}$, $68 \pm 2\text{g}$, and $64 \pm 2\text{g}$ of *E. citriodora*, *C. winterianus*, and *C. martini*, respectively. The extractions were performed at 16°C and 70 bar (condition 1) and 25°C and 160 bar (condition 2). A static period of 60 minutes was used. The experiments were performed in duplicate. The extraction yields varied with material and operational conditions. For the *E. citriodora* extract the yield was 0.31% for condition 1 and 0.68% for condition 2. The yield was 0.45% and 0.99% for conditions 1 and 2 for *C. winterianus*. The yields for the *C. martini* extracts were 0.07% and 0.20% for conditions 1 and 2, respectively. The SC extraction yields were less than that of steam distillation (*E. citriodora*: 2.50%; *C. winterianus*: 2.15%; *C. martini*: 1.39%) for all samples. The chemical composition of the extracts was analyzed by GC-MS.

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Article Outline

[Author comments, 25 May 2001 16:09:20, by M. Angela Meireles](#)

Supplementary material.

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This paper shows the composition of essential oils from *C. martini*, *C. winterianus* and *E. citriodora* obtained by SFE and conventional techniques. The results also shows the fractionating effects of SFE.

Supplementary material.

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Supercritical Extraction of Essential Oils of *Cymbopogon martini*, *Cymbopogon winterianus*, and *Eucalyptus citriodora* with Carbon Dioxide

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ABSTRACT. The extraction of essential oils of *Eucalyptus citriodora*, *Cymbopogon winterianus* and *Cymbopogon martini* has been studied using supercritical carbon dioxide as solvent. The raw materials were dried at room temperature ($30 \pm 2^\circ\text{C}$) for 5 days. The dried leaves were cut in pieces of 1cm (*Eucalyptus citriodora*) or 3cm of length (*Cymbopogon winterianus* and *Cymbopogon martini*). The fixed bed was packed with $87 \pm 1\text{g}$, $68 \pm 2\text{g}$, and $64 \pm 2\text{g}$ of *E. citriodora*, *C. winterianus*, and *C. martini*, respectively. The extractions were performed at 16°C and 70 bar (condition 1) and 25°C and 160 bar (condition 2). A static period of 60 minutes was used. The experiments were performed in duplicate. The extraction yields varied with material and operational conditions. For the *E. citriodora* extract the yield was 0.31% for condition 1 and 0.68% for condition 2. The yield was 0.45% and 0.99% for conditions 1 and 2 for *C. winterianus*. The yields for the *C. martini* extracts were 0.07% and 0.20% for conditions 1 and 2, respectively. The SC extraction yields were less than that of steam distillation (*E. citriodora*: 2.50%; *C. winteria-*

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nus: 2.15%; *C. martini*: 1.39%) for all samples. The chemical composition of the extracts was analyzed by GC-MS.

KEYWORDS. Supercritical extraction, essential oils, *Cymbopogon martini*, *Cymbopogon winterianus*, *Eucalyptus citriodora*, chemical composition, citronella, eucalyptus, palmarosa

INTRODUCTION

Essential oils are currently used as raw material for the perfumery, pharmaceutical, cosmetic, and food industries. Brazil, because of its large agricultural resources, can potentially become one of the major worldwide producers of essential oil.

Among the many species cultivated in Brazil, from which essential oils are extracted for local markets and for export, we may mention eucalyptus (*Eucalyptus citriodora*, Hook) and grass of the *Cymbopogon* gender, and among them, citronella (*Cymbopogon winterianus*, Jowitt) and palmarosa (*Cymbopogon martini*, (Roxb.) are of major importance. In 1998 Brazil exported 214.6, 8.9 and 1.5 tons of essential oils from eucalyptus, palmarosa and citronella, respectively⁴.

The gender *Eucalyptus* belongs to the *Myrtaceae* family and has about 800 species/subspecies. They occur naturally in Australia, and *Eucalyptus citriodora* is primarily grown in Brazil and Congo⁵. The citronella is largely cultivated in Asia and in some countries of Central America. There are two kinds of citronella, which produce essential oils of different chemical characteristics. The most important is known as Java and is obtained from *Cymbopogon winterianus*, while the Ceylon type is obtained from *Cymbopogon nardus* Rendle.

The palmarosa grows abundantly in India, where it is called “Rushua” or “Rosha”. Like the citronella essential oil, the palmarosa oil is used in the cosmetic industry, particularly in

soaps, to which it imparts a pronounced and lasting rose note. The main components of this essential oil are geraniol, citronellal, and citral⁷. The essential oils eucalyptus, citronella and palmarosa are raw materials of high commercial value. The geraniol and citronellal are used in the preparation of important reactants to the perfume industry such as citronellol, hidroxicitronellal, nerol, menthol, etc².

In previous works with these species^{5,6} grown at Barra Bonita, state of São Paulo, Brazil, the composition of the extracts obtained by steam distillation (SD) and CO₂ extraction (SCFE) were reported. The operational conditions were 70 bar and 16°C. To compare the yield and chemical composition of the extracts it was used gas chromatography and mass spectrometry. The yields for the SD methodology were higher (1.45% for the eucalyptus, 1.50% for the citronella, and 1.67% for the palmarosa) than the yields of SCFE (0.74% for the Eucalyptus, 0.34% for the citronella, and 0.14% for the palmarosa). The palmarosa essential oils obtained by the two methodologies presented different relative proportion of the main chemical compounds: linalool (3.07% SD, 1.55% SCFE); geraniol (84.90% SD, 87.88% SCFE); geranyl acetate (6.02% SD, 3.58% SCFE); caryophyllene (2.56% SD, 3.97% SCFE). For the citronella essential oil, the relative proportions of the main components were citronellal (37.04% SD, 18.28% SCFE); citronellol (23.51% SD, 24.30% SCFE); and geraniol (29.58% SD, 24.85% SCFE). The other components were monoterpenes and sesquiterpenes (6.45% SD, 27.57% SCFE). The eucalyptus essential oil has the same behavior. With the steam distillation technique, the citronellal (42.22%) and citronellol (40.16%) summed together 83.38 % of the extract, and mono and sesquiterpenes composed the rest (16.62%). For SCFE the main components summed together 58.83% of the extract (32.20% citronellal and 21.63 % citronellol), and the rest summed together 41.17%.

In this work the effects of the temperature and pressure on the extract yields and on the composition are presented for the essential oils of eucalyptus, citronella and palmarosa, at the operational conditions of 16°C and 70 bar (condition 1) and 25°C and 160 bar (condition 2).

MATERIALS AND METHODS

Materials. The raw material utilized, eucalyptus (*Eucalyptus citriodora*, Hook), citronella (*Cymbopogon winterianus*, Jowitt) and palmarosa (*Cymbopogon martini*, (Roxb.)), were collected at the experimental farm of the Agronomic Institute of Campinas in March of 1999 and were dried at room temperature ($32\pm 2^\circ\text{C}$) for 5 days. The materials were collected manually and cut to the size of 1 cm for the eucalyptus and 3 cm for the citronella and palmarosa.

Extraction Line. The essays were performed using an extraction unity similar to the one used by Monteiro¹ (Figure 1). This equipment has one CO₂ reservoir (CS) (99% purity, White Martins Industrial Gases), two stainless steel jacketed cylinders (White Martins Co., model 316 LSS DOF3A200, USA), 200 cm³ each, and kept at -10°C , acting as heat exchangers and used to refrigerate the solvent (TC1 and TC3). The refrigerating medium was an aqueous solution of ethylene glycol (90%) re-circulating in a thermal bath (BT1) (Polyscience, model 9510, USA). Two heat exchangers (TC2 and TC4) made of two coils (316 Stainless steel 6 m length and 1/8" nominal diameter) were also used. TC2 was immersed in the thermal bath BT1 and TC4 was connected to a second thermal bath (BT2) that utilizes water as a refrigerating liquid. This last heat exchanger elevates the solvent temperature to the extraction temperature and acts as a surge tank to avoid pressure oscillations at the pump head. An HPLC pump (Bb) (Thermoseparation Products, model Constametric 3200 P/F, USA) controls the system's pressure. The extraction column (CE) is made of stainless steel (0.43 m of length, 3.3×10^{-3} m of diameter, Brazil) and contains an

extraction cell (stainless steel, 316 SS, 0.375m of length, 2.73×10^{-3} of diameter, Brazil). For operational easiness, the extraction cell is completely removable from the extraction unit. The system pressure was controlled with 3 manometers (M1, M2 and M3) (TERBRASMA, model 2541, 100 ± 1 bar, Brazil, and RECORD, model 1554, 500 ± 1 bar, Brazil). To control the system's temperature, it was used a thermocouple (iron-constantan) adapted to a temperature register with 5 entries (Robert Shaw, model T4WM, USA), connected to the extraction column and pump inlets and outlets. The system had also two filters (F) (0.2 and 20 μm , Swagelok, model SSF-4F, TFN-986, USA), needle-type valves (V1 to V6, Autoclave Engineers, model 316 SS, USA), V7 (Detroit, model 1864BSS4HC, USA). The flow fine control was made by a micrometering valve (VM, Autoclave Engineers, Series 10VRM 2812, USA). This valve was covered with a heating tape (length of 1.3 m, Fisaton, model 5, Brazil) to avoid line freezing during the gas expansion). The valve temperature control was made by a thermocouple connected to a temperature sensor (DinaSense, model 2156-40, USA). The collector Flask (CC, 20 cm^3 volume) and a glass column (CP, 0.6 cm internal diameter, 15 cm length), filled with adsorbent material (2g of Porapak Q, 80-100 mesh, waters Associated Inc. USA), were connected at the valve outlet, for the capture of the volatile compounds. The flow meter (MV) was described by Monteiro et. al¹⁰.

FIGURE 1. Flow sheet of the experimental unit.

Extraction Procedure. The raw materials were set inside the extraction cell. The fixed bed was packed with 87 ± 1 g, 68 ± 2 g, and 64 ± 2 g of *E. citriodora*, *C. winterianus*, and *C. martini*, respectively. The extraction cell was adapted at the extraction column, and the system was pressurized with the pump. The CO_2 was left in contact with the material at the operational conditions for the static period of one hour, and then the valves V7 and VM were opened and the extraction

process began. The essential oils extractions were conducted at two operational conditions (16°C, 70 bar and 25°C, 160 bar) with replications. Samples of the extracts were collected after 2 hours of extraction (CC), during the system decompression (DESC₁) and in the Porapak column assembled at the solvent exit, as described by Monteiro et al.¹⁰ (CP). In order to avoid losses in the extraction unit due to the high viscosity of the citronella and of the eucalyptus extracts, two decompressions of the system were used, producing two samples (DESC₁ and DESC₂). The collector flasks and the capture column were immersed in an ice bath to prevent losses of the extract's most volatile components. After each sampling, the flasks were weighted, sealed, and stored in a domestic freezer (Brastemp, model 410, Brazil). The extract's mass was quantified using an analytical balance (± 0.0001 g, Sartorius, model A200S, USA). The solvent flow was continuously measured and controlled throughout the extraction and varied from 2.18 to 2.50 g CO₂/min.

The species' essential oils were also obtained by steam distillation in a modified Moritz equipment for the same extraction time employed for the SCFE.

Chemical Composition of the Extracts. The analysis of the chemical composition of the essential oils was made using gas chromatography - mass spectrometry, (GC-MS, Shimadzu, QP 5000), ionization voltage 70 eV, equipped with capillary column DB-5 (J & Wiley Scientific, 30 m x 0.25mm x 0.25 μ m), injector and detector at 240°C and 230°C, helium as carrier gas, flow rate 1.7 mL/min, split mode 1/30, sample injection volume 1 μ L, temperature program of 50°C – 280°C at 4°C/min. The identification of the chemical constituents was made by comparison of the mass spectra of the substances with the equipment's (Nist 62 libr.) databank and literature data^{8,1}.

The quantitative analysis was conducted using gas chromatography (GC-FID, Shimadzu, model GC-17 AAF CBM101), detector temperature 280°C, operating at the same conditions of

the GC-MS. To quantify the main essential oil components, the external standard method, described by Collins³, was used. The following standards were employed: citronella (P.A. Sigma, Lot: 84F0436), citronellol (95%, Cutrale Ltda., Brazil), geraniol (P.A. Sigma, Lot: 17H3508) and geranyl acetate (66%, Dierberger Óleos Essenciais S.A. Ind., Brazil). The standards were diluted in ethyl acetate and seven solutions were prepared with replicates.

RESULTS AND DISCUSSIONS

The results demonstrate (Table 1) that at 160 bar and 25°C, the masses of the samples collected during the first two hours of extraction of citronella, eucalyptus, and palmarosa were smaller than the other samples (CP, DESC₁, and DESC₂). The same behavior was observed for the citronella at 70 bar and 16°C, while for the palmarosa and eucalyptus the CC samples were larger for the same condition.

Raising the pressure from 70 to 160 bar and temperature from 16 to 25°C resulted in higher yields for all the species (Table 2). Nevertheless, the yields were smaller than the ones obtained by the conventional method of steam distillation (2.5% for the eucalyptus, 2.15% for the citronella and 1.39% for the palmarosa).

TABLE 1. Mass of Extracts (g) obtained for the various extract samples.

TABLE 2. Average yields relative to the feed (wet raw material).

The citronella extracts (CC, DESC₁, DESC₂) presented a similar phytochemical profile for the two operational conditions studied, but with different relative proportions of the chemical constituents (Table 3). The main constituents were geraniol, citronellal, citronellol, and γ -

gurjunene. The geraniol relative proportion for the tree samples (CC, DESC₁, DESC₂) at 160 bar was 78.65%, while at 70 bar it was 87.09%. The proportion of citronellal, citronellol, and γ -gurjunene were 61.78%, 37.71%, and 37.24% for 160 bar and 45.42%, 42.93%, and 38.45% at 70 bar. The content of the major substances in the oil is shown in Table 4.

TABLE 3. Composition of the extracts of citronella (area %).

TABLE 4. Content of the Major Substances in the citronella Extracts (mass %)

Table 5 shows the chemical composition of the eucalyptus oil. A comparative analysis of the chromatograms of the extract for the two conditions reveals that samples DESC₁ and DESC₂ have heavier compounds at 70 bar. At 160 bar and 25°C, the citronellol is the most abundant component in CC sample (58.77%), while for the DESC₁ sample the citronellal is the main constituent (76.67%). At 70 bar and 16°C, the citronellol is the most abundant component at all samples (50.96%, CC; 40.54%, DESC₁; 39.4%, DESC₂); the same was observed for the citronellal (39.39%, CC; 22.25%, DESC₁; 18.77%, DESC₂). The content of the main compounds of the eucalyptus oil is in Table 6.

TABLE 5. Composition of the extracts of eucalyptus (area %).

TABLE 6. Content of the major substances in the eucalyptus extracts (mass %)

The extracts of the palmarosa (Table 7) showed different chemical compositions at the two operational conditions studied. The monoterpenes geraniol, geranyl acetate, and linalool are the most abundant constituents. At 160 bar, the CC sample shows exclusively geraniol (100%)

while at 70 bar it was 91.29%. The relative proportion of monoterpenes, linalool, geraniol, geranial, carvyl acetate and geranyl acetate at the extract DESC₁ summed up 81.40%, while at 70 bar it was 58.18%. The content of the major substances present in the palmarosa oil is shown in Table 8.

TABLE 7. Composition of the extracts of palmarosa (area %).

TABLE 8. Content of the major substances in the palmarosa extracts (mass %)

CONCLUSIONS

The increase in pressure from 70 to 160 bar and in temperature from 16 to 25°C resulted in an increase of the yield for the Palmarosa (*Cymbopogon martini* (Roxb.)), Citronella (*Cymbopogon winterianus*, Jowitt), and Eucalyptus (*Eucalyptus citriodora*, Hook) extracts. For the three species the chemical composition of the extracts varied with the operational conditions. At 70 bar and 16°C SCFE the extracts had larger relative percentage of heavier compounds. The vegetable material was not subjected to a drying process, as is usually recommended¹⁰. The fact that the SD yields were always larger than the SCFE yields indicates that the presence of water negatively influences the SCFE process. This is probably due to the interactions between the water, the essential oils components, and the cells walls. Therefore, the pretreatment of the vegetable material, which includes drying at controllable conditions, is a very desirable procedure.

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TABLE 1. Mass of extract (g) obtained for the various extract samples

Fraction	Citronella		Eucalyptus		Palmarosa	
	160 bar, 25°C	70 bar, 16°C	160 bar, 25°C	70 bar, 16°C	160 bar, 25°C	70 bar, 16°C
CP	0.2624	0.0975	0.2624	0.0975	0.0359	0.0242
CC	0.0571	0.0377	0.0571	0.0377	0.0059	0.0154
DESC ₁	0.2800	0.1838	0.2800	0.1838	0.0798	0.0036
DESC ₂	0.1144	0.0957	0.1144	0.0957	-	-
Total	0.7139	0.4147	0.7139	0.4147	0.1586	0.0432

CP: sample captured in the Porapak column; **CC:** sample obtained after 2 hours of extraction; **DESC₁:** mass of extract obtained during the first decompression; **DESC₂:** mass of extract obtained during the second decompression; **Total:** sum of CP, CC, DESC₁, and DESC₂.

TABLE 2. Average yields relative to the feed (wet raw material)

	Yield, % (mass of extract / mass of feed)		
	Citronella	Eucalyptus	Palmarosa
70 bar, 16°C	0.45	0.31	0.07
160 bar, 25°C	1.00	0.68	0.20
Steam Distillation	2.15	2.50	1.39

TABLE 3. Composition of the citronella extracts (area %)

Substance	Operational Condition					
	160 bar e 25°C			70 bar e 16°C		
	CC	DESC ₁	DESC ₂	CC	DESC ₁	DESC ₂
α -pinene	0.38	Tr	Tr	0.22	0.29	Tr
Citronellal	23.37	21.90	17.37	10.53	16.09	18.80
Citronellol	15.80	11.04	10.87	18.53	14.26	10.14
Geraniol	31.99	23.71	22.95	37.10	29.39	20.60
Citronellyl acetate	2.13	2.34	2.06	1.47	1.78	1.57
Eugenol	2.30	2.25	2.00	2.49	2.18	1.78
Geranyl acetate	2.49	2.50	2.24	1.86	1.79	1.50
γ -elemene	0.95	1.13	0.87	0.75	0.90	0.95
Aromadendrene	3.85	4.69	3.94	3.09	3.99	3.82
α -humulene	3.66	7.45	5.27	2.47	5.64	6.25
γ -gurjunene	10.15	14.70	12.39	11.05	15.17	12.23
Hydrocarbons	0.50	2.99	11.93	6.22	2.60	14.91
Total Identified	97.57	94.70	91.89	95.78	94.08	92.55

Tr = traces

TABLE 4. Content of the major substances in the citronella extracts (mass %)

Substance	Operational Condition					
	160 bar e 25°C			70 bar e 16°C		
	CC	DESC ₁	DESC ₂	CC	DESC ₁	DESC ₂
Citronellal	10.32	7.02	7.35	7.72	7.12	6.26
Citronellol	5.32	0.48	1.74	12.90	4.13	0.58
Geraniol	15.82	5.14	8.02	32.70	13.06	4.54
Eugenol	3.02	3.72	3.49	1.29	2.82	3.49
Geranyl acetate	1.65	2.33	2.09	1.01	1.97	2.47

Table 5. Composition of the eucalyptus extracts (area %)

Substance	Operational Condition				
	160 bar and 25°C		70 bar and 16°C		
	CC	DESC ₁	CC	DESC ₁	DESC ₂
t-3-hexen-1-ol	-	-	-	-	-
Isopulegol	0.56	1.22	0.57	Tr	Tr
Citronellal	34.07	70.67	39.39	22.25	18.77
Citronellol	58.77	19.75	50.96	40.54	39.40
Caryophyllene	2.99	4.16	3.64	2.84	3.91
Sesquiterpenes	1.49	0.94	1.70	2.63	2.98
e-phytol acetate	0.70	0.89	Tr	6.72	6.36
Saturated Aliphatic Hydrocarbons	-	0.65	Tr	6.36	5.70
Total Identified	98.58	98.28	96.26	81.34	77.12

TABLE 6. Content of the major substances in the eucalyptus extracts (mass %)

Substance	Operational Condition				
	160 bar and 25°C		70 bar and 16°C		
	CC	DESC ₁	CC	DESC ₁	DESC ₂
Citronellal	18.74	41.79	24.92	2.36	2.80
Citronellol	40.57	17.03	39.13	15.05	17.40
Caryophyllene	0.42	0.16	0.13	1.40	0.95

TABLE 7. Composition of the palmarosa extracts (area %)

Substance	Operational Condition			
	160 bar , 25 °C		70 bar , 16 °C	
	CC	DESC ₁	CC	DESC ₁
Linalool	Tr	1.70	0.40	Tr
Geraniol	100	74.50	91.29	55.87
Geranial	-	0.33	-	Tr
Carvyl acetate	-	0.57	0.42	Tr
Geranyl acetate	Tr	4.93	2.18	2.31
Caryophyllene	-	0.38	Tr	1.75
β-farnesene	-	0.41	0.76	4.43
Saturated aliphatic hydrocarbons	-	5.70	-	5.81
Total Identified	100	87.88	95.06	70.17

TABLE 8. Content of the major substances in the palmarosa extracts (mass %)

Substance	Operational Condition			
	160 bar , 25°C		70 bar , 16°C	
	CC	DESC ₁	CC	DESC ₁
Linalool	0.00	9.17	9.98	0.00
Geraniol	1.96	39.82	39.04	0.72
Geranyl acetate	0.00	2.45	4.86	5.83
β-Caryophyllene	0.00	3.73	3.90	3.74

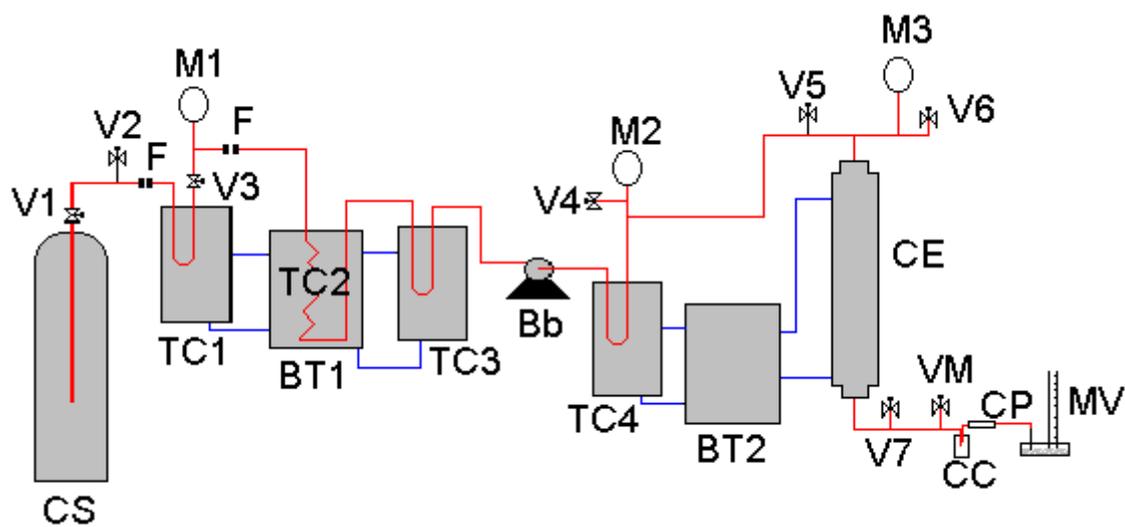


FIGURE 1. Flow sheet of the experimental unit.