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Feasibilty study of the technology of fatty coriander oil complex processing

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ABSTRACT

Organoleptic, physico-chemical and spectro-photometric properties for unrefined and refined fatty coriander oil are determined in the present study. Physiologically active substances (unsaturated fatty acids, betacarotenes, phospholipids and chlorophylls) were identified. Fatty acid and acylglycerol content in the whole fractions (liquid and semi-solid) was determined. Characterization and comparison of properties of refined oil obtained with different techniques was performed. Developed scientifically justified method of FCO refining extraction with 96% ethyl alcohol allows decreasing the content of FFA from 8,5% to 0,2%.

It was indicated that extraction refining of FCO with ethyl alcohol as extraction agent provides elimination of FFA and do not interfere the content of natural chlorophyll in the refined oil. Application of new proposed technology provided generation of two products – "Coriander petrozelin" and coriander salad dressing oil. Technological properties of obtained products were defined. As obtained data indicates, content of SOO* acylglycerols in separated semi-solid fraction was 5.58 times higher than content of SLO* acylglycerols.

In accordance with obtained results equipment and technological scheme of complex FCO processing, comprising refining and fractional crystallization as the main stages of the process.

Introduction

Modern concept of healthy human nutrition requires new approach for oil and fat production technologies. Implementation of such products is very promising and can be beneficial for chronic diseases prevention, supporting of human health and life expectancy. Natural oils, containing unsaturated fatty acids (FA) play an important role in food chain, human nutrition and widely used in everyday products [1,2].

Fatty coriander oil (FCO) is potentially a new product that could extend the assortment of oil and fat products. Coriander have been planted during recent years in forest-steppe and steppe zones of Ukraine. Among Ukrainian oil and fat producers there is only one facility, producing FCO, which is employing the press method. This facility is specialized on production of high quality coriander essential oil; FCO is diverted and marketed as nonfood byproduct. At the same time FCO is a source of unsaturated oleic acids (up to 85%), which is mainly represented by petroselinic acid (up to 75%). Petroselinic acid differs from oleic in that it has higher melting point (up to 30 °C) [3–5]. This property of FCO can be used for derivation of semi-solid fraction, which could be free from *trans*-isomers, almost free from saturated FA (<4%) and can extend the assortment of special purpose oils.

Effective use of unrefined FCO in food production technologies is limited because it contains high amounts of free fatty acids (FFA), formed during essential oil derivation from coriander seeds (acid number up to 17 mg of KOH per gram and of free fatty acids content at rate of 8,5%) due to specialties of derivation technology. Traditional way of refining (extraction of FFA) is not feasible for oils as it results in generation of large amounts of waste. Typical process of FFA removal from oils, which have high acid number (alkaline and distillation neutralization) is accompanied with high rate of gross fat loss [6–8].

According to the analysis of the literature data [9] Russian scientists had developed the technology of FCO refining, which comprises following technological stages: hydration, neutralization, rinsing, drying, bleaching, filtration, desodoration.

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Reduction of the amount of wastes is one of the priority tasks for increasing the overall refining efficiency [10–12], making thus feasible the task of development of FFA elimination technique using selective solvents, i.e. by extractional refining [13,14].

Preliminary study of the process of FCO refining with ethyl alcohol was conducted in the National Technical University « KPI». Experimental study of the technological process of FCO extractional refining comprised: reasoning of the selective solvent choice, study of solvent properties on extraction rate of FFA from FCO, evaluation of the impact of extractional refining different technological parameters (temperature, hidromodule and duration of the process) on the effectiveness of FFA removal from the given media [15]. Next task set in the scope of this study was the development of the technology of fractioning crystallization for refined FCO. As it known, FCO composition is represented mainly by tripetroselinic acylglycerols – 41,80% and dipetroselino-mono-oleic (or linoleic) acylglycerols (PPeO, PPeL, POPe, PLPe) – 43%, monopetroselinic acylglycerols (PPeO, PPeL, POPe, PLPe) – 7% [16,17]. Due to the specific of FA composition of FCO until the present time it was processed only through the unselective hydrogenation for low iodine solid fat formation, which is used as a raw material for stearic acid production. However, this specialty of FCO composition makes it possible to apply physical process of it's modification – fractioning, which does not require chemical treatment [18].

As the next stage of the study, trial derivation of the whole (semi-solid) fraction of FCO was performed.

At the first time the whole fraction “Coriander petroselin” with melting point 19–25°C, that has high content of unsaturated FA (96.7%) and does not contain trans isomers, which can be used as a special purpose fat is obtained [19].

Organoleptic, physicochemical and spectrophotometric properties were studied, fatty acid and acylglycerol composition was defined during study and equipment – technological scheme of the process of the complex FCO refining was developed.

Data and analytical methods

Defining of organoleptic and physic-chemical indicators of FCO

Taste, transparency and flavor of FCO was determined organoleptically – according to the Standard “Oils. Determination of flavor, color and transparency”. Determination of the color number was conducted according to SSU (State Standard of Ukraine) 4568:2006 “Oils. Methods for the determination of the color number”.

Determination of acid number – according to SSU (State Standard of Ukraine) 4350:2004 “Oils. Method for the determination of the acid number (ISO 660:1996, NEQ)”.

Determination of acid number – according to SSU (State Standard of Ukraine) 4570:2006 “Vegetable fats and Oils. Method for the determination of the acid number”.

Determination of anisidine number according to SSU (State Standard of Ukraine) ISO 6885–2002 “Fats and oils animal and vegetable. Determination of the anisidine number”.

Determination of the iodine number – according to SSU (State Standard of Ukraine) ISO 3961:2004 “Animal fats and vegetable oils. Determination of the iodine number”.

Determination of refraction indicator – according to SSU (State Standard of Ukraine) ISO 6320–2001 “Fats and oils animal and vegetable. Determination of the refraction”.

Determination of Phosphorus-containing compounds – according to SSU (State Standard of Ukraine) 7082:2009. “Oils. Methods for the determination of the bulk part of phosphorus-containing compounds”.

Determination of beta-carotene – according to SSU (State Standard of Ukraine) EN 12823–2:2006 “Food products. Determination of vitamin A by the liquid chromatography method with high separation ability. Part 2. Determination of b-carotene content”.

Determination of the bulk part of non-fat inclusions – according to SSU (State Standard of Ukraine) ISO 663–2003 “Animal and vegetable fats and oils. Determination of non-dissolved inclusions content.” (ISO 663:2000, IDT).

Determination of the bulk part of the moisture and light compounds – according to SSU (State Standard of Ukraine) 4603:2006 “Oils. Methods for the determination of the mass part of the moisture and light compounds”.

Determination of the content of unsaponified substances according to SSU (State Standard of Ukraine) ISO 3596:2004 “Animal and vegetable fats. Determination of the content of unsaponified substances. Method employing the extraction with diethyl ethere.” (ISO 3596:2000, IDT).

Determination of spectrophotometric indicators of FCO

Chlorophylls, carotenoids and FFA content in the initial and refined FCO was determined on the spectrophotometer Spectrod M 40 (Germany). For determination of absorption spectrum of initial and refined FCO, measurements in ultraviolet diapason were performed with solutions of FCO and hexane with concentration 10 mg/ml in cuvettes 2 mm; for the measurements in the visible part of the spectrum, solutions with concentration 40 mg/ml were prepared and 10 mm cuvettes were used for the measurements.

Determination of fatty acid and acylglycerol composition of refined FCO products

Products of fractional crystallization of FCO, in particular liquid and semi-solid fractions were analyzed with gas-liquid chromatography method for determination of their fatty acid and acylglycerol composition.

Fatty acid composition of FCO was determined according to SSU ISO 5509–2002 “Animal and vegetable fats — Preparation of methyl esters of fatty acids. (ISO 5508:2001, IDT)”. Preparation of samples was performed as following: sample of the oil was dissolved in 2 ml of heptane; 200 μl of methanol base solution was added with micropipette and mixed during 5–10 min; 1 g of sulfur monohydrate of sodium was added while shaking. Upper layer, which contained methyl esters of FA was separated after. Solution of methyl esters of fatty acids was analyzed by chromatography (ISO 5508:1900, IDT).

Detection of FA was performed on the gas chromatograph “Chrom-5” (Czech republic). The length of the chromatography column was 3.5 m with external diameter 5 mm and internal diameter 3 mm. The temperature of thermostat, evaporator and detector was, accordingly 190 °C, 230 °C and 250 °C. Argon was used as a gas carrier, introduced sample volume was 0,2–0,3 μl. Methyl esters of FA “Merck” were used as external standards.

Acylglycerol composition of the products of fractioning of FCO was determined in accordance with SSU ISO 5509:2002 “Fats and oils animal and vegetable. Preparation of methyl esters of FA (ISO 5509:2000, IDT).

Acylglycerol composition of samples was identified employing gas-liquid chromatography method, using gas chromatograph Hewlett Packard HP-6890 with capillary column HP 88 (88% cyanopropyl acryl-polysiloxane, Agilent Technologies) with length 100 mm, internal diameter 0,25 mm and thickness of the stationary phase 0,2 μm.

Conditions were following: flow rate of the gas carrier – 1,2 ml/min, flow separation coefficient – 1:100, temperature of the evaporator – 280 °C, temperature of the detector – 290 °C,

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temperature regime of the column – gradual heating from 60 °C to 230 °C. Acylglycerols were identified according to the duration of their retention in comparison with retention time for the known samples. The content of particular acylglycerols was calculated in percent from the gross weight. Registration and processing of chromatograms was performed with HP ChemStation software.

Results and analysis

Comparative characteristics of the composition of FCO samples, obtained by different techniques

Comparative characteristics of the samples of refined FCO, obtained by different techniques is presented in Table 1.

High FFA content (16.47 mg KOH/g or 8.5%) in coriander vegetable oil can be explained with high moisture content (0.62%) in initial product. In this connection twin-screw extrusion seems more prospects for the future [20–24]. There were some problems with the storage conditions of fruits before hydraulic pressing. Indeed, when fruits are stored with too high moisture content, this can lead to in-situ hydrolysis phenomena, thus contributing to the transformation of tri-glycerides in di- and mono-glycerides and even FFA. Meantime, it can be assumed, that obtained refined FCO has satisfying organoleptic and physico-chemical qualities, which meet existing standards for edible oils.

Content of substances detected by spectrophotometry in FCO samples

For detection of chlorophylls presence, carotenoids content in refined FCO and verification of FFA elimination from unrefined FCO, spectrophotometric study was performed. Absorption spectrums for initial and refined FCO samples in ultraviolet diapason are depicted in Fig. 1.

He analysis of absorption spectrum of the initial and refined FCO solutions in ultraviolet diapason of the spectrum (200–350 nm), few absorption lines were detected. In particular, asymmetric wide lines (upper – initial FCO, lower – refined FCO) in the diapason 200–250 nm with maximum at 210–215 nm (D = 1,22 and D = 0,98 accordingly). These lines are associated with absorbtion

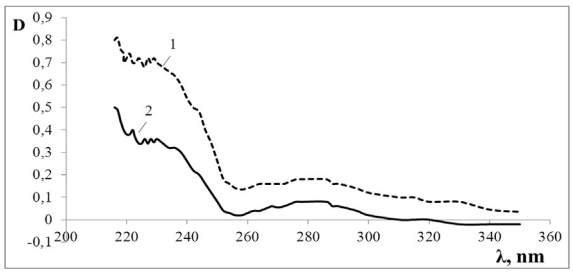

Fig. 1. Spectrums of absorbtion of the solutions of initial and refined LCO in ultraviolet part (concentration 10 mg/ml in 2 ml cuvette): 1 – initial FCO; 2 – refined FCO.

of saturated and unsaturated FA (spectrum of polyunsaturated FA with isolated double bonds does not differ from spectrum of monounsaturated FA, as the presence of isolated ethylene bond is crucial for the chromoform. Symmetrical wide line from 260 nm to 310 nm can be related to triacylglycerols' absorbtion in general. The decrease of optical density of refined FCO with 280 nm wavelength down to D = 0,06 can be explained by the solubility of FFA in the extraction agent and their elimination during the technological process of FCO extraction refining. Spectrums of absorption of the solutions of initial and refined FCO in the visible part of the spectrum are depicted on Fig. 2.

During the analysis of the spectrum of absorption for initial and refined FCO solutions two lines in the visual part of the spectrum

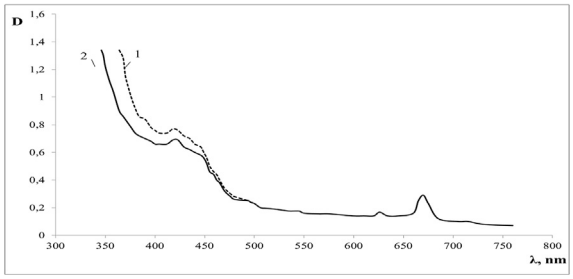

Fig. 2. Spectrums of absorbtion of the solutions of initial and refined LCO in the visible part (concentration 40 mg/ml, cuvette 10 mm): 1 – initial FCO; 2 – refined FCO.

Table 1
Comparative characteristics of organoleptic and physic-chemical indicators of FCO.

Indicator	Characteristics of the indicator				
	FCO*	FCO**	FCO***	FCO****	FSO*****
Taste	Bittery taste	Flavorlessoil taste	Flavorless oil taste	Flavorless oil taste	Flavorless oil taste
Smell	strong smell	odorless	odorless	odorless	odorless
Acid number, mg KOH/g	16,47	0,40	0,36	0,38	0,25–0,60
Peroxide number, mmole ½O/kg	12,52	2,34	–	4,02	6,0–10,0
Anisidine value, units.	3,1	–	–	2,3	–
Iodine number, gI_2/100 g	107,18	–	–	107,20	–
Refraction indicator at 20°C	1,4720	–	–	1,4631	–
Color number, mg of iodine	69	20–30	8	25	12
Beta-carotene (polyvitaminA), mg/kg	16,1	–	–	12,8	–
Bulk part of phosphorus containing substances recounted for stearooleolecithins,%	0,53	absent	absent	0,32	absent
Mass part of the moisture and light substances, %	0,62	0,10	–	0,11	0,10
Bulk part of non-fat impurities, %	0,15	absent	–	0,15	absent
Bulk part of unsaponified substances, %	1,5	2,0	0,98	1,5	–

FCO* - unrefined oil obtained with hydraulic press using.
FCO** - obtained by the basic refining [9].
FCO*** - obtained by the basic refining with use of specific acid (patent №:2101336 RF).
FCO**** - obtained by the extractional refining with ethyl alcohol [14].
FSO***** - regulatory requirements for refined sunflower oil (State standard of Ukraine SSU4492:2005).

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360–750 nm were detected. In particular, two simple, non-symmetrical, wide lines in the diapason 400–480 nm with maximum at 420 nm are related to absorption of carotenoids. Decreasing of the optical density of refined FCO at 420 nm to D = 0,67 can be explained by particular solubility of the carotenoids, for example xanthophylls, in ethyl alcohol in the technological process of extractional refining of FCO.

It appeared that in the diapason 500–750 nm two lines merge into one with two symmetrical peaks of absorption. Peak at 640 nm with optical density D = 0,15 can be related to chlorophyll *b*, peak at 660 nm with D = 0,225 – to chlorophyll *a*. Ratio of chlorophylls *a*/b was 1,5. Thus, it was defined, that extractional refining of FCO provides elimination of FFA, but do not interfere natural chlorophylls content in refined FCO.

Fatty acid composition of the whole fraction of the FCO

The study of the fatty acids composition of the liquid and semi-solids fractions, obtained after the dry fractioning of FCO was performed. Obtained data of the chromatography study are presented in Table 2.

As Table 2 indicates, general content of FA, in particular, petroselin-oleic (C18:1) and linoleic acid (C18:2) in separated liquid fraction of FCO was at the rate of 96.53%. It is necessary to emphasis that the column used during GC was inappropriate for separating both isomers, i.e. oleic (C18:1n-9) and petroselinic (C18:1n-12) acids. An adequate column would have contributed to separate oleic and petroselinic acids, thus leading a more precise quantification [20].

It should be noted, that for the first time trans isomers of linoleic acid (C18:2n6tr) were detected in the composition of the liquid fraction. Gross content of FA, in particular petroselin-oleic (C18:1) and linoleic (C18:2), in separated semi-solid fraction FCO was 96.44%.

Acylglycerol composition of the whole fraction of FCO

The study of acylglycerol composition of the liquid and semi solid fractions, obtained after dry the fractioning of FCO was performed as described hereinabove.

Acylglycerol composition of derived liquid and semi-solids fractions of FCO was analyzed using high-temperature gas-liquid chromatography. Data on the chromatography study of the samples are presented in Table 3.

As Table 3 indicates, gross content of acylglycerols, in particular, SOO* and SLO* in separated liquid fraction of FCO was 88,04% with the ratio 2,14:1, i.e. acylglycerols SOO* content was twice higher than for SLO* acylglycerols. The content of acylglycerols in separated semi-solid fraction of FCO reached 100%. Mainly FCO solid fraction acylglycerols were represented by SOO* and SLO*, their gross content was 92.10%.

Development of the equipment and technological scheme of FCO complex processing

Universal equipment and technological scheme of refined FCO and technical fractions production employing extractional refining and fractional crystallization methods was developed on the basis of performed studies results and theoretical generalization of obtained results. A hypothetical technological scheme of the complex processing of FCO is presented in Fig. 3.

According to the given scheme, technological process of FCO extractional refining is performed as follows: reactor *R1*, as marked on the scheme, is fed with unrefined FCO and ethyl alcohol. In the reactor *R1* the feed is mixed and heated up to +78 °C and held at this temperature during 10 min, then directed to the decanter *D1*, where dynamic separation of the mixture into two layers (upper – alcohol, lower – purified FCO) is performed.

Purified FCO from the decanter by means of the centrifugal pump *BH1* is directed to the reactor *P1* and ethyl alcohol once again is added. This technological operation is repeated twice.

Upper layer – alcohol extract, from the decanter *D1* is directed by gravity to the holding tank *E1* and re-processed after (ethyl alcohol is distilled in the rectification unit and returned to the process and the extract – FA concentrate is considered as this stage as a special purpose raw material for sale).

Refined FCO from the reactor *D1* is directed by gravity into the tank *E2* and than pumped out by centrifugal pump into the evaporation unit *V1* where FCO is dried at the temperature 80 °C to evaporate ethyl alcohol residuals.

Dried FCO is directed to the *E3* holding tank by gravity. Ethyl alcohol vapor from the evaporation unit *V1* is directed to the heat exchanger *T1*, where it condensed and forwarded after to the vessel *B1* by gravity and ethyl alcohol is pumped out of the vessel by centrifugal pump and returned into the technological process of the extractional refining into *P1* reactor.

For obtaining of the special purpose fractions from the refined FCO, the technological process of the fractional crystallization is employed, which is performed as following: from the tank *E3* refined FCO is pumped into crystallization chamber *K* with centrifugal pump *BH3*. In the crystallization chamber *K* oil is mixed and chilled to the temperature of +5 °C with the pace 1 °C per hour.

Oil chilling is supported by circulation of the cool water through the crystallization chamber water jacket. Fraction separation is performed by the filtration of the crystalized oil on the filer *FC1*.

Fractions mixture from the crystallization chamber *K* is directed to the filter *FC1* by gravity where by means of the vacuum pump the separation of the liquid oil fraction is performed which is collected in the internal filer chamber while semi-solid fraction “Coriander petroselin” is remained on the surface of the belt filter.

Table 2
Fatty acid composition of liquid and semi-solid fraction of FCO.

No	Liquid fraction		Semi-solid fraction	
	Fatty acid	Content of the fatty acid, %	Fatty acid	Content of the fatty acid, %
1	Palmitic acid $C_{16:0}$	3,07	Palmitic acid $C_{16:0}$	2,34
2	Stearic acid $C_{18:0}$	0.67	Stearic acid $C_{18:0}$	0.93
3	Oleic acid $C_{18:1n9}$+ + Petroselinic acid $C_{18:1n12}$	79,43	Oleic acid $C_{18:1n9}$+ + Petroselinic acid $C_{18:1n12}$	87,87
4	Linoleic acid $C_{18:2n6tr}$	0,29	Linoleic acid $C_{18:2\ n6tr}$	0,00
5	Linoleic acid $C_{18:2n6c}$	15,74	Linoleic acid $C_{18:2n6c}$	8,57
6	Linolenic acid $C_{18:3n3}$	0,24	Linolenic acid $C_{18:3n3}$	0,11
7	Arachidic acid $C_{20:0}$	0,11	Arachidic acid $C_{20:0}$	0,00
8	Gadoleic acid $C_{20:1}$	0,46	Gadoleic acid $C_{20:1}$	0,18

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Table 3
Acylglycerol composition of the liquid and semi-solids fractions of FCO.

No	Retention time, min	Liquid fraction		Semi-solid fraction	
		Acylglycerol *	Acylglycerol bulk content, %	Acylglycerol *	Acylglycerol bulk content, %
1	16,32	POS	2,84	POS	2,81
2	16,66	POS	4,34	POS	2,07
3	17,79	PLO	5,22	PLO	2,14
4	21,15	SOS	0,54	SOS	0,87
5	21,77	SOO	36,61	SOO*	62,02
6	22,23	SOO	20,62	SOO*	16,09
7	22,48	SLS + OOO	3,01	SLO*	1,39
8	23,75	SLO	26,81	SLO*	12,60

*(O) – Acyl: cis 6-7 C18:1 – Petroselinic acid.
*(O) – Acyl: cis 9-10 C18:1 – Oleic acid.

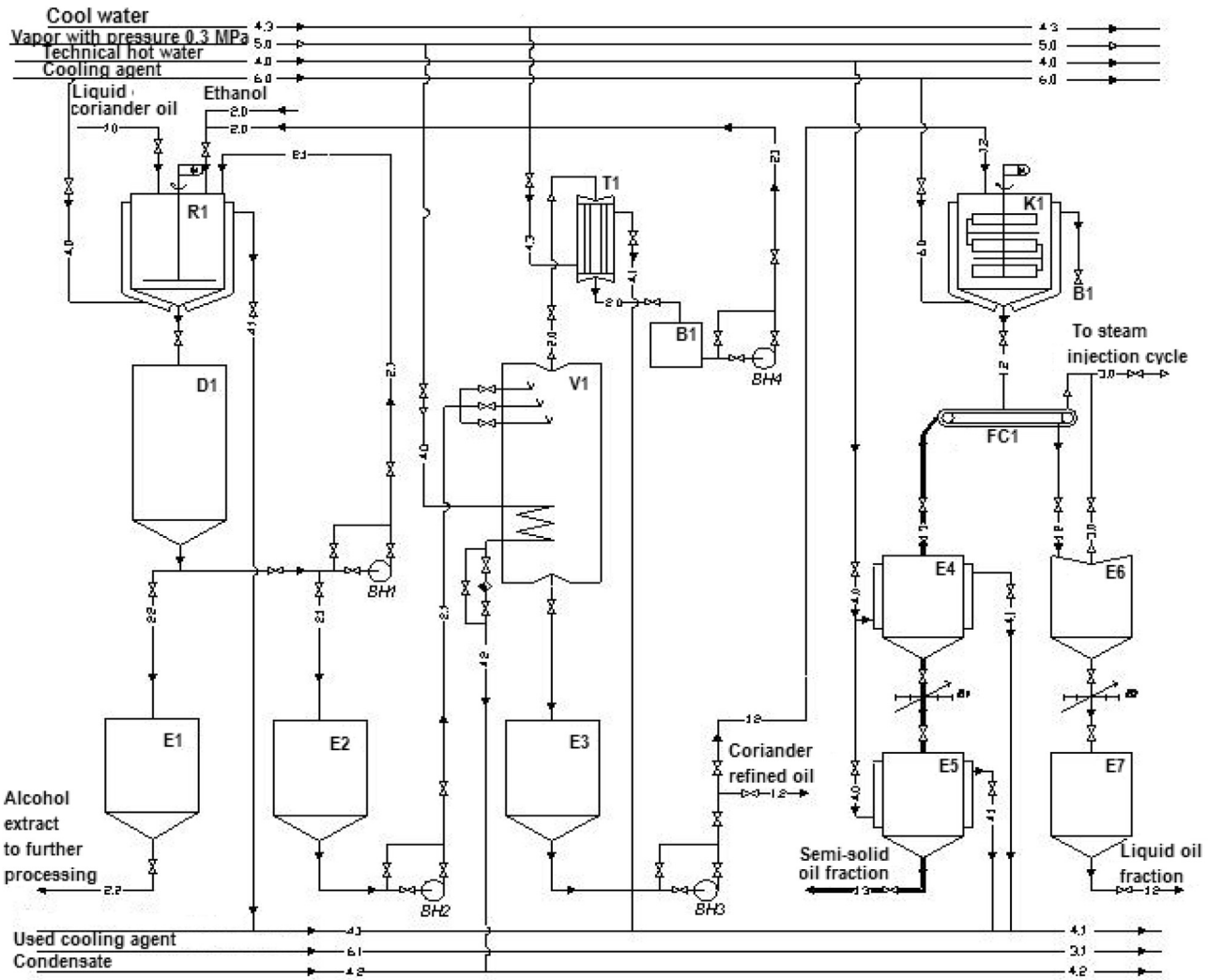

Fig. 3. Equipment and technological scheme of the complex processing of FCO.
R1 – reactor, *E 1–7* – tanks, *D1* – decanter, *BH 1–4* – centrifugal pumps, *V* – evaporator, *K1* – crystalizator, *T1* – heat recuperator, *B1* – tank, *FC1* – belt filter, *BL1-2* – balances.

Sediment is removed with the knife from the belt and forwarded to the tank *E4*, which is equipped with water jacket where hot water is circulating. Liquid "Coriander petroselin" is pumped through the balances BL1 and collected in the tank *E5* and forwarded to the packing from the tank.

Liquid fraction while withdrawn from the filter *FC1* by gravity into vacuum vessel E6 through the balances BL2 and collected in the tank *E7* and forwarded to the packing stage from there.

According to the developed technological scheme of complex FCO processing liquid and semisolid fractions were obtained. Semi-solid fraction FCO – "Coriander petroselin" is meeting the requirements for baking fat SSU 4336, liquid fraction – "FCO dressing" was recommended for the use of refined coriander vegetable oil as a food supplement, at a maximal level of 600 mg per day [4].

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Conclusions

We have studied and identified organoleptic and physic-chemical properties of obtained samples of refined FCO (according to the new developed technology of extractional refining with ethyl alcohol) and compared those with properties of FCO obtained by other methods.

Developed scientifically justified method of FCO refining extraction with 96% ethyl alcohol allows to decrease the content of FFA from 8,5% to 0,2%.

It was indicated that extraction refining of FCO with ethyl alcohol as extraction agent provides elimination of FFA and do not interfere the content of natural chlorophyll in the refined oil.

Application of new proposed technology provided generation of two products – "Coriander petrozelin" and coriander salad dressing oil. Technological properties of obtained products were defined. As obtained data indicates, content of SOO* acylglycerols in separated semi-solid fraction was 5.58 times higher than content of SLO* acylglycerols.

In accordance with obtained results equipment and technological scheme of complex FCO processing, comprising refining and fractional crystallization as the main stages of the process.

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