CHEMICAL AND PHYSICOCHEMICAL CHARACTERIZATION OF VEGETAL LIPID FRACTIONS ISOLATED FROM SWEET PEPPER (*CAPSICUM ANNUUM*) AND CHILLI PEPPER (*CAPSICUM FRUTESCENS*)

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Abstract. *The main purpose of this paper was to obtain vegetal lipid fractions from processing by-products in order to superior exploit of some material bearers of food utilities integrated in horticulture and / or agro-food processing. Isolation of seed oil from sweet pepper (Capsicum annuum) and chilli pepper (Capsicum frutescens) was done by extraction solid / liquid solvent using an extract equipment Soxhlet type, followed by purification and characterization of chemical and physical - chemical of lipid vegetal fractions (acidity, esterification, saponification, unsaturated index).*

Keywords: *fatty acids, chilli peppers, sweet peppers, acid index, esterification index, hydroxyl index*

INTRODUCTION

Superior acids isolated and specifically purified through repeated liquid/liquid extractions, after gas-chromatographic characterisation were processed into monitored structured functional lipids.

Raw materials required for achievement of experimental part [**sweet peppers** (*Capsicum annuum*) and chilli peppers (*Capsicum frutescens*)] it has been harvested from the Western Romania.

No significant difference in the chemical composition of the raw material from different geographical areas consists in the high degree of unsaturations of superior acids as such or directed by the ω3 and / or ω6 systems.

This paper can provide a real direction of integrated ecological recovery of by-products mentioned above,in full agreement with environmental European and world treaties.

Basic colloidal characterization aims to focus future research directions for optimal technological and agro-alimentary horticultural processing (and not only) among which these structures through synergic cumulus of defining competences of the poly-unsaturated superior acids ***(1Δ; 2Δ; 3Δ)***, of "homogeneous" derivatised poly-ethylene-oxi chains (***PEO***) with monitored degree of oligomerisation ***(n = 3 – 18)*** (JIANU ET AL., 2014, 2010).

Future research aims to access oligomeric chains ***PEO*** processed through Williams synthesis adapted in order to its role in the global "colloidal fingerprint" of the studied conjugate ***PEG_n-L (2R';R) (R';2R)*** (JIANU, 2014).

Lipids natural and/or synthetic PEGylated represents for food, cosmetics, pharmaceuticals, drugs, physical proprieties, physical-chemical, physiological, nutritional and unique chemical proprieties. ***O`Brien (1998)*** consider that their functionality in "***host***" food products in general, is influenced by: ***the composition*** and distribution of ***superior fatty*** acids; the ***crystallisation mode***; its ***own flavor*** and their stability; the associated mode with other nonlipid molecules (water, antioxidants, antifoaming agents, chelating agents, colorants,

crystallization inhibitors, preservatives, emulsifiers, etc.), and resistance to food, pharmaceutical and/or drug processing (JIANU, 2007, 2008).

MATERIALS AND METHODS

Materials: seed material of sweet pepper (*Capsicum annuum*) and chilli pepper (*Capsicum frutescens*)); petroleum ether (p.f.=30-60°C)

Methods **(NEGREA, 2011):**

Hot extraction of lipid fractions from seeding material (seeds, fruits, berries)

In a porcelain mortar are weighed (via difference) to ***with precision of 0.01 g,*** plant material seeds 10 - 15 g, is added 15 g Na_2SO_4 anhydrous and is transfer quantitatively using a spatula in a ***extraction cartridge (Soxhlet) .***

It cleanses the mortar, pestle and spatula with a soaked cotton swabin solvent, and subsequently introducing the cellulosic cleaningmaterial in the cartridge. It cover with clean cotton and extraction unit is mounted in the heating system.

Enter the solvent ***(extraction petrol, petroleum ether with p.f. = 30 - 60°C or hexan)*** until is prime the complete siphoning, when it added 50 mL of solvent.At reflux temperature is carried out ***8…10 siphoning /hour***, ***6 hours***, after the cartridge is removed and is recovered the solvent. Miscela clear with no impurities, filter, additional quantitative calibrated into a flask.

Vegetal lipid fraction purified, is dried exhaustively for ***30 minutes at 105°C***, in the oven, is cooled in desiccator and weigh. Is repeated if necessary drying and weighing operations to constant weight.

In order to facilitate the removal of solvent traces in the recovery operation of the extraction solvent, is bubbled through flask a stream of nitrogen ***(8 – 10 mL/minute).***

Vegetal lipid fraction (seed) of samples is evaluated with the follow relation:

$$\textbf{\textit{Vegetal lipid fractions}} = \frac{m_2 - m_1}{m_3} \cdot 100(\%)$$

where:

$\boldsymbol{m_1}$ – empty flask weight, (***g***);

$\boldsymbol{m_2}$ – flask weight with vegetal lipid fraction, (***g***);

$\boldsymbol{m_3}$ – weight of the sample taken for evaluation, (***g***).

RESULTS AND DISCUSSIONS

The resulting crude vegetalacids fraction ***after full recovery*** of the solvent extraction were assessed chemical and physico-chemical properly.

Table 1

The chemical and physico-chemical property of chilli and sweet pepper

	Chilli pepper	***Sweet pepper***
Unsaturations degree	***96.72%***	***97.3%***
The content of saturated fatty acids	***3.19%***	***2.7%***
Average molecular weight	***225.278***	***281.013***
Iodine index	6·253,8g	3·253,8

From the analysis chromatograms in the gas phase ***(figura 1)*** two major information can be obtained:

- ✓ ***high degree of unsaturation (96.72%*** chilli pepper*; 97.3%* sweet pepper);

✓ ***equivalent weight (average molecular weight, $\overline{M}$) of superior acids.***

In all studied variants unsaturatation is provided, in varying proportions, by the superior acids: ***oleic (C_{18};1Δ) (M=282); linoleic (C_{18};2Δ) (M = 280) and linolic (C_{18};3Δ) (M = 278).***

The content in saturated superior acids aprox. ***3.19% C_{16}*** chilli pepper ***(p_c)*** și ***2.7% C_{16}*** sweet pepper ***(p_s)*** doesn't change ***footprint*** of vegetal lipid fractions rich in ***ω_3 acids*** with the increased interestin the composition of functional foods in the last decades.

It is noted that in saponified lipid fraction of chilli and sweet pepper predominate acids ***C_{18};1Δ, in proportion of 57%*** respectively ***83%)***

Evaluation of equivalent weight (average molecular weight, $\overline{M}$) was made with relations below adapted to gaschromatographic composition of each lipid fractions saponified:

(p_c) $\overline{M}\,p_c$ ***= 0.133 · 278 + 0.675 · 280 + 0.159 · 282 + 0.0319 · 256 = 225.278***

(p_s) $\overline{M}\,p_s$ ***= 0.134 · 280 + 0.830 · 282 + 0.027 · 256 = 281.013.***

Aciditity evaluation index of fatty acids superior, free formed after total acid hydrolysis (HCl) of vegetal lipid fractions extracted in the solid / liquid system was done with the relation:

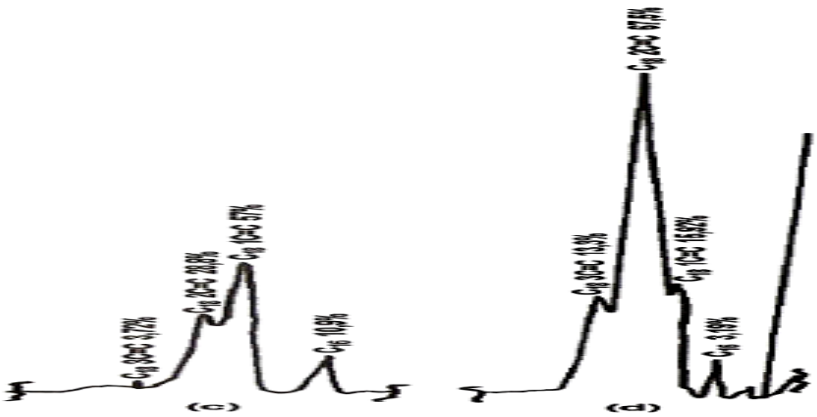
1 mole free fatty acid ----------► 1 mole potassium hydroxide

$\overline{M}_m$ (g) ----------► 56000 mg KOH

$\overline{M}_{co}$

1 g product evaluated ----------► I_A

$$I_A \text{ (acid index)} = \frac{56000}{\overline{M}} \quad \text{[mg KOH/g product evaluated]}$$



Evaluation of the equivalent molecular weight of the free superior acids [C_{16}, respectively C_{18} (1,2,3Δ)] of vegetal lipid fractions gas chromatographic separated: (a) 281.012; (d) 225.278

Fig. 1. Gas chromatogram of the superior fatty acid methyl esters free isolated by total acid hydrolysis and cesterification with BF_3/CH_3OH fromvegetal saponifiable lipid fractions [chilli pepper (p_c) and sweet peppers (p_s)] stationary phase Chromosorb W (80/100 mesh), carrier gas (N_2) (10 mL/min), temperature gradient 5°C/minute, flame ionization detector

In the processing and interpretation of experimental data can be found structures with different ester functional groups. Differentiating between their indices esterification could be achieved because the average molecular weights (weight equivalent).

The experimental values obtained have confirmed the accuracy of reasoning and using of "cross"analytical evaluation systems. Thus, ***were simultaneously correlated the experimental results provided by acidity, esterification, hydroxyl indices etc.***

For ***esterification indices*** close to the theoretical values was natural that the acidity indices respectively hydroxylated structures involved in esterification record low values or is zero.

Simultaneous corroborating of these indicators has allowed and further specification processing of yields, but also the purity of the structures in question (obviously closely monitored after purification).

Assessment of iodine index of structures with high degree of unsaturation was also an analytical "cross" assessment method because through data corroborating experiments with the gas phase chromatograms allowed certification of equivalent weight values ***(of average molecular weight, $\overline{M}$)*** of different accesed structures.

1 mol unsaturated fatty acids ($\overline{M}$) ---------- q moles I_2 (253.8 g)

M_m (6Δ) (225,278) $6 \cdot 253{,}8$ g iodine

M_{co} (3Δ) (281,012) $3 \cdot 253{,}8$ g iodine

100 g unsaturated evaluated product ---------- I_{iodine}

$$I_{iodine} \text{ (iodine teoretical index)} = \frac{q \cdot 253.8}{\overline{M}} \cdot 100 \text{ [mg KOH/g evaluated product]}$$

Ponderal medium value

In each case (chilli pepper, sweet pepper) the participation proportion of unsaturated superior acids ***1Δ; 2Δ; 3Δ*** but overall the differences corresponds to the q value above.

In the case are accessed simultaneously two different saturated chains ***(2R') (chilli pepper/sweet pepper)*** iodine will have real access conferred by the weighted average value and proportion of mutual participation.

Assessment of iodine index is justified through the intention to confirm / invalidate any adverse change in the cleavage structure.

CONSCLUSIONS

The analysis of chromatograms in the gas phase can be obtained two major information:

- ✓ ***high degree of unsaturation (96.72%*** chilli pepper*; 97.3%*** sweet pepper);
- ✓ ***equivalent weight (average molecular weight, $\overline{M}$) of superior acids.***

In the saponified lipid fraction of chilli peppers and sweet peppers predominate acids ***C_{18}; 1Δ, in proportion of 57%*** respectively ***83%).***

The experimental values obtained have confirmed the accuracy of reasoning and using of "cross" analytical evaluation systems. Thus, ***were simultaneously correlated the experimental results provided by acidity, esterification, hydroxyl indices etc.***

Therefore characterized vegetable lipid fractions (saponifiable) were used in simultaneous and sequential transesterification of 2,5 - diacetyl - isosorbide.

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