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VALORIZATION TECHNOLOGIES OF SOME HORTICULTURAL PLANTS WITH MEDICINAL USAGE

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ABSTRACT *– The extraction by maceration in hydro-alcoholic solutions is the oldest method of using alcoholic spirits. Since ancient times, there were concerns on studying the stability of these preparations and finding fakes. We have been studying this problem for many years. The paper presented a specific evolution in time (5 years), according to assortment, from the moment of preparation and during the many years of preservation in airtight glass containers. The extract (g/100 ml) has varied either by diminution (six cases) or by higher values of fresh macerates, but there were two cases of stability, too. The content of soluble dry matter has increased in most of macerates, the cases of unchanged values or diminution being in minority. The colour parameters (luminosity, chromatic, the two coordinates of colour and shade) have evolved differently, giving the possibility of identifying in time the extract authenticity of every species. The determinations done in dynamics have shown the evolution of chemical composition of hydro-alcoholic concentrates, achieved during 5 years. We have also made 18 spectra, in order to study comparatively, the two types of macerates, the fresh ones, prepared in 2007, and the others, preserved for many years (determined in both 2006 and 2007). Determinations are a basis of repeatability and possible identification.*

Key Words: hydro-alcoholic extracts, medicinal and aromatic plants, macerate stability, non-reducing extract, colour parameters

REZUMAT - Tehnologii de valorificare a unor plante horticole cu utilizări medicinale. *Extracţia prin macerare în soluţii hidroalcoolice reprezintă cea mai veche metodă de utilizare a distilatelor alcoolice. Din perioadele mai vechi au existat preocupări pentru urmărirea stabilităţii acestor preparate şi pentru depistarea falsurilor. Autorii au preocupări de mai mulţi ani privind problematica în cauză. Lucrarea identifică o evoluţie caracteristică în timp (5 ani), în funcţie de sortiment, din momentul preparării şi pe parcursul păstrării multianuale*

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în recipiente de sticlă, etanşe. Extractul (g/100 ml) a variat, fie prin diminuare (6 cazuri), fie prin valori superioare maceratelor proaspete, dar au existat şi două cazuri de stabilitate. Conţinutul în substanţă uscată solubilă a crescut la majoritatea maceratelor, cazurile de valori neschimbate sau de diminuare fiind minoritare. Parametrii de culoare (luminozitatea, cromaticitatea, cele două coordonate ale culorii şi tonalitatea) au evoluat foarte diferit, dând posibilitatea unei identificări în timp a autenticităţii extractelor fiecărei specii. Determinările efectuate în dinamică au reflectat evoluţia compoziţiei chimice a concentratelor hidroalcoolice, realizate de-a lungul perioadei de timp de 5 ani. S-au realizat şi 18 spectre, pentru a putea urmări, comparativ, cele două tipuri de macerate, cele proaspete, preparate în 2007, şi cele păstrate pe o perioadă de mai mulţi ani (determinate atât în 2006 cât şi în 2007). Determinările reprezintă o bază de repetabilitate şi de posibilă identificare.

Cuvinte cheie: extracte hidroalcoolice, plante medicinale şi aromatice, stabilitatea maceratelor, extract nereducător şi parametrii de culoare

INTRODUCTION

The maceration of plants is the oldest method of using alcoholic spirits. In the IX-X century B.C., these technologies became known by doctors and alchemists. Called *el-ixir* in the Arabian language and *liqueur* or *tinctura* in the Medieval Latin, they have evolved until nowadays, either as pharmaceutical preparations or as alcoholic drinks (liqueurs). From the experience of liqueur makers, it is well known that some macerates are more active as freshly prepared, while others, only after a period of maturation. It is also known that there are many fakes and substitutes for the expensive products. (Agopian, 1975; Grigorescu, 1987; Ionescu, Savopol 1977).

Previous studies (Beceanu et al., 1999; Beceanu et al., 2004; Beceanu, Hobincu, 2006; Râpă et al., 1998) have stimulated us to continue the analysis of these products, by approaching new species and checking their possible modifications in time.

MATERIALS AND METHODS

In this paper, we have investigated 17 species of aromatic and/or medicinal plants (Crăciun et al., 1977; Grigorescu et al., 1986; Potlog, Vinţan, 1985). The vegetal material was obtained in two stages. The first stage was during 2001 – 2003, with analyses made in 2006. In the second stage, the material was sampled in 2006 and analysed in 2007.

The sampled vegetal material was packed in plastic bags and transported for processing. The plants have been introduced in containers and then we have added alcohol for obtaining a certain concentration for the extraction of the active principles. After these operations, the containers were closed airtight and stored in the storehouse of the Technology of Horticultural Product Department.

The alcoholic extractive solutions are pharmaceutical liquid preparations, obtained by extracting some vegetal products by means of alcohol. Maceration consists in the treatment of the minced vegetal product with the quantity of prescribed solvent and its

keeping for a determined time at the normal temperature. The factors influencing extraction are the nature of the vegetal product, product humidity, mincing degree, nature of solvent, environment reaction (pH), ratio between the vegetal product and solvent, duration of maceration and temperature (Ionescu, Savopol, 1977; Rădoiaş et al., 1988).

No	Scientific denomination	Popular denomination	Plant organ used	1st study phase	2nd study phase
1.	*Achilea millefolium*	Yarrow	flowers and leaves	2001	2006
2	*Acorus calamus*	Calamus	roots and rhizomes	2001	2006
3	*Angelica archangelica*	Angelica	roots and rhizomes	2001	2006
4	*Amomum cardamon*	Cardamom	fruits	2001	2006
5	*Carum carvi*	Caraway	fruits	2001	2006
6	*Ceratonia siliqua*	Carob	fruits	2001	2006
7	*Coriandrum sativum*	Coriander	fruits	2001	2006
8	*Gențiana lutea*	Gentian	roots and rhizomes	2001	2006
9	*Hysopus officinalis*	Hyssop	flowers and leaves	2001	2006
10	*Iris germanica*	Iris	roots and rhizomes	2001	2006
11	*Juglans regia*	Walnut	fruits	2002	2006
12	*Juniperus communis*	Juniper	fruits	2001	2006
13	*Mentha x piperita*	Mint	flowers and leaves	2001	2006
14	*Ocimum basilicum*	Basil	flowers and leaves	2001	2006
15	*Salvia officinalis*	Sage	flowers and leaves	2001	2006
16	*Sambucus nigra*	Elder	flowers, leaves and fruits	2003	2006
17	*Zingiber officiale*	Ginger	roots and rhizomes	2001	2006

The maceration extracts contain a complex of substances, some of them with net physiological action, while others are inactive. Substances are extracted selectively, and in the extractive solution, a new ratio among components appears (Beceanu et al., 2003; Grigorescu et al., 1986; Potlog, Vințan, 1985).

The intimate mechanism of all these modifications is highly complex. The physical or chemical determinations may establish quite precisely, the quantity of components considered active, but in other cases, only the biological determinations may appreciate the activity of preparations (Beceanu et al., 2004; Ionescu, Savopol E., 1977; Rădoiaş et al., 1988).

The extraction of the soluble substances from the solid raw material through maceration in solvent is called diffusion. By diffusion, they understand the phenomenon of interpenetration of liquids or gases, in contact with each other. By the contact of two solutions containing a dissolvent substance at different proportions or of a solution with a pure solvent, even without stirring, the dissolved substance has the tendency to equal its concentration in the entire liquid volume. In this case, the movement of the substance goes towards the homogenization of the mixture that is towards the equalization of the

concentration. This phenomenon has its explanation in the kinetic theory of the solution formation (Beceanu et al., 2004; Ionescu, Savopol, 1977; Rădoiaş et al., 1988).

They use alcohol as a solvent. Small capacity glass or enameled containers are also used. The solution is filtered for removing the residue that is extracted with a new quantity of solvent. The solutions are reunited and let to rest for a long time for maturation. After maturation, they are filtered and filled up to the prescribed concentration (Beceanu et al., 2004).

Physical-chemical analyses of the samples under study (Beceanu et al., 2004; Coşofreţ, 1997). Density is determined by means of the electronic densimeter (reference method) following the steps: decarboxylation, in order to eliminate the influence of the dissolved gases, then after a correct washing of the densimetric cell with the working sample, we initiate the determination, the device achieving a temperature correction up to the value of 20.000°C. The results may be reproduced up to ± 0.0001 g/cm^3. The total acidity consists in the titration (neutralization of acids) of the extract sample with a solution of normality nitrate hydroxide, in the presence of the pH-meter, as an indicator of the equivalence point (pH=7), after the preliminary removal of the carbon dioxide.

Alcoholic concentration – the sample is decarboxylated, in order to eliminate the influence of the dissolved gases, then we distillate it with a preliminary neutralization of the volatile acids. The distillate is collected in the collecting flask, where we have measured the sample to equate the measured volumes. The measurement is done by means of the electronic densimeter with which we measure at least three readings.

The non-reducing extract was determined according to STAS 184/3-70 for alcohol-based products with traces of extract, using a gravimetric method with the calculated reproducibility of ± 0.0001 g/100 ml.

The colour parameters of extracts were determined by UV-VIS Analytik Jena Specord 200 spectrophotometer and tested by CIE Lab76 method (luminosity L – the psychometrical clarity ranges between 0 black-opaque and 100 colourless-transparent; a – coordinate of complementary colours red (+) - green (–); b - coordinate of complementary colours yellow (+) – blue (–); C – the chrome or saturation of colour shows that the extract may have a more or less pure colour; H – the colour hue corresponds to the angle, expressed in sexagesimal degrees).

RESULTS AND DISCUSSION

In *Tables 1 and 2*, we showed the evolution of the physical-chemical parameters of the hydro-alcoholic extracts. One might notice for the year 2006 the presence of a minimum and a maximum at the percentage extracts for cardamom and carob, which could be correlated to the relative density. As for the analyses made in 2006, we could not show clearly the evolution of the extract, since the high alcoholic concentration has influenced density and, implicitly, the extract.

The values presented in *Tables 3-5* were in accordance with the visual aspect, characteristic to the plant and the colour of the vegetal part, which was exposed to maceration (Crăciun et al., 1976; Grigorescu et al., 1986; Potlog, Vinţan, 1985).

The study carried out in these two years is a completion, by the comparative chromatic analyses, which have shown the specificity of these extracts. The chromatic parameter **L** (luminosity) could highlight efficiently the evolution of extracts from the viewpoint of stability of the chemical combinations in time. For example, we mention the extracts of angelica and carob, presenting a concentration in time of the extracted compounds.

Table 1

Physical-chemical characteristics of the extracts analysed in 2006

Sample	Extract (g / 100 mL)	Extract (%)	Relative density to water	Alcohol % v/v	Soluble dry matter (°Bx)
Angelica	1.06	1.09	0.97389	13.30	10.4
Basil	1.45	1.56	0.93033	26.50	19.4
Cardamom	0.10	0.12	0.84918	40.44	22.0
Caraway	1.17	1.40	0.83618	38.86	23.6
Yarrow	1.67	1.77	0.94506	23.48	17.8
Coriander	0.41	0.49	0.82769	34.36	22.2
Ginger	0.37	0.43	0.84856	39.18	22.0
Gentian	1.06	1.29	0.82783	43.60	22.2
Hyssop	0.92	1.09	0.84935	37.32	21.6
Juniper	2.51	3.01	0.83596	38.96	24.0
Mint	0.70	0.83	0.84176	44.58	22.0
Green walnuts	3.45	3.67	0.94239	24.65	20.8
Calamus	1.07	1.29	0.83353	40.65	22.8
Carob	5.23	6.26	0.83618	42.59	23.2
Sage	0.30	0.36	0.84417	36.25	20.8
Elder flower	1.35	1.64	0.82384	42.68	21.8
Elder fruit	3.58	3.66	0.98038	13.88	13.6
Iris	1.97	2.36	0.83690	42.48	22.4

Table 2

Physical-chemical characteristics of the extracts analysed in 2007

Sample	Extract (g / 100 mL)	Extract (%)	Relative density to water	Alcohol % v/v	Soluble dry matter (°Bx)
Angelica	1.98	2.03	0.97591	27.97	13.4
Basil	1.32	1.34	0.98784	14.87	7.0
Cardamom	0.23	0.24	0.96657	28.88	10.5
Caraway	0.49	0.51	0.96583	30.83	13.0
Yarrow	1.17	1.22	0.98040	40.31	14.2
Coriander	0.49	0.51	0.96419	32.05	12.8
Ginger	1.26	1.29	0.97240	28.45	13.8
Gentian	1.74	1.79	0.97267	28.76	13.4
Hyssop	1.61	1.65	0.97381	28.62	10.4
Juniper	1.41	1.46	0.97157	29.21	12.8
Mint	1.40	1.43	0.98462	17.07	8.0

Sample	Extract (g / 100 mL)	Extract (%)	Relative density to water	Alcohol % v/v	Soluble dry matter (°Bx)
Green walnuts	2.49	2.55	0.97803	28.44	14.2
Calamus	0.89	0.95	0.93699	49.69	18.0
Carob	10.27	10.17	1.01154	26.47	20.4
Sage	1.32	1.36	0.97234	28.14	12.6
Elder flower	1.43	1.48	0.97120	29.67	12.8
Elder fruit	9.98	10.07	0.99330	41.16	13.8
Iris	4.69	4.67	1.00734	10.51	8.4

By analysing the absorption spectra and all the chromatic parameters, we have noticed that certain extracts, such as basil, coriander, juniper, hyssop and mint had a possible tendency to concentrate only in certain classes of compounds like carotenoids or chlorophylls. Because the quantity of anthocyans was high in the elder extract, in time we noticed a net differentiation with a possible increase of phenolic compounds that gave the extract a more intense red coloration. In contrast with what we knew about anthocyans in fermentation, the colour was more stable and intense at extraction, without unpleasant phenomena of polymerization ($[\text{-p-}]_n$) or poly-condensation ($[\text{-p}]_x\text{-}[\text{c-}]_y$) (Beceanu et al., 2003).

Table 3

Chromatic characteristics of the extracts analysed in 2006

Sample	Luminosity L	Colour coordinates		Chromaticity C	Tonality H
		a red(+) - green(-)	b yellow(+) - blue(-)		
Angelica (2001)	91.58	-1.22	36.08	36.11	-88.07
Basil (2001)	70.02	2.46	73.7	73.74	88.09
Cardamom (2001)	96.58	-3.22	18.86	19.13	-80.32
Caraway (2001)	79.36	3.49	73.62	73.7	87.29
Yarrow (2001)	77.98	6.5	70.52	70.82	84.73
Coriander (2001)	88.43	-9.84	40.35	41.53	-76.29
Ginger (2001)	90.25	-1.23	53.17	53.19	-88.67
Gentian (2001)	92.72	-4.32	49.18	49.37	-84.99
Hyssop (2001)	68.48	-1.97	51.08	51.12	-87.79
Juniper (2001)	80.64	-0.6	66.48	66.48	-89.48
Mint (2001)	18.38	6.07	27.35	28.02	77.48
Green walnuts (2002)	21.73	29.61	36.69	47.15	51.09
Calamus (2001)	93.25	-0.95	32.04	32.05	-88.31
Carob (2002)	86.97	-2.98	50.55	50.63	-86.62
Sage (2001)	89.58	-5.79	40.81	41.22	-81.93
Elder flower(2003)	80.07	-5.9	52.64	52.97	-83.61
Elder fruit (2003)	5.80	30.9	9.23	32.25	16.63
Iris (2001)	9.24	36.31	15.3	39.4	22.85

We might speak of a favorable long-term maceration, by analysing the evolution of the chromatic parameters, parallel with the dry extract for some extracts, such as juniper and walnut (*Juniperus communis* and *Juglans regia*). There were still exceptions in the extracts of carob (*Ceratonia siliqua*), elder (*Sambucus nigra* -fruits) and iris (*Iris germanica*), where a long-term process proved to be unfavorable (the dry extract diminished) or even inexistent (the extract value did not change significantly) for coriander (*Coriandrum sativum*) and elderflower (*Sambucus nigra*) (Crăciun et al., 1977; Grigorescu et al., 1986; Potlog, Vințan, 1985).

Table 4

Evolution of the chromatic characteristics of the extracts analysed in 2006, and repeated in 2007

Sample	Luminosity L	Colour coordinates		Chromaticity C	Tonality H
		a red(+) - green(-)	b yellow(+) - blue(-)		
Angelica (2001)	94.34	-2.65	30.54	30.65	-85.04
Basil (2001)	48.51	7.03	55.88	56.32	82.83
Cardamom (2001)	96.20	-3.30	20.81	21.07	-80.98
Caraway (2001)	76.45	7.9	80.03	80.42	84.36
Yarrow (2001)	75.79	9.74	74.91	75.54	82.59
Coriander (2001)	80.00	0.13	69.12	69.12	89.89
Ginger (2001)	88.87	-1.28	56.05	56.07	-88.7
Gentian (2001)	92.24	-1.53	28.93	28.97	-86.96
Hyssop (2001)	35.74	6.72	44.64	45.14	81.44
Juniper (2001)	91.59	-8.54	32.42	33.52	-75.24
Mint (2001)	16.78	7.5	28.52	29.48	75.27
Green walnuts (2002)	23.73	38.27	40.86	55.98	46.88
Calamus (2001)	92.59	-0.43	35.88	35.88	-89.32
Carob (2002)	88.02	-2.74	50.14	50.21	-86.86
Sage (2001)	89.73	-5.95	42.6	43.01	-82.05
Elder flower (2003)	73.86	3.5	70.94	71.03	87.18
Elder fruit (2003)	8.33	37.03	14.36	39.72	21.2
Iris (2001)	5.32	29.68	9.18	31.07	17.19

Table 5

Chromatic characteristics of the extracts analysed in 2007

Sample	Luminosity L	Colour coordinates		Chromaticity C	Tonality H
		a red(+) - green(-)	b yellow(+) - blue(-)		
Angelica	84.86	3.42	49.65	49.77	86.06
Basil	27.25	36.09	46.49	58.86	52.18
Cardamom	93.91	-0.31	15.39	15.39	-88.84
Caraway	97.23	-2.07	13.14	13.31	-81.05

Sample	Luminosity L	Colour coordinates		Chromaticity C	Tonality H
		a red(+) - green(-)	b yellow(+) - blue(-)		
Yarrow	91.6	-4.9	47.56	47.81	-84.12
Coriander	93.6	-1.59	26.18	26.23	-86.53
Ginger	79.47	9.81	64.35	65.09	81.33
Gentian	67.96	19.54	78.85	81.24	76.08
Hyssop	82.57	4.01	42.21	42.2	84.59
Juniper	98.59	-1.09	6.94	7.03	-81.09
Mint	81.49	5.65	64.96	65.21	85.03
Green walnuts	65.47	36.4	109.53	115.42	71.62
Calamus	90.93	0.11	28.17	28.17	89.78
Carob	62.16	31.72	93.56	98.79	71.27
Sage	74.00	14.68	69.64	71.17	78.09
Elder flower	91.06	-2.28	36.11	36.19	-86.39
Elder fruit	1.22	8.85	2.11	9.1	13.38
Iris	86.2	-4.19	18.56	19.02	-77.27

The spectra done by means of UV-VIS Analytik Jena Specord 200 spectrophotometer have a unique and specific aspect, giving the possibility of acknowledging macerates (extracts) and finding out fakes (*Figures 1-18*).

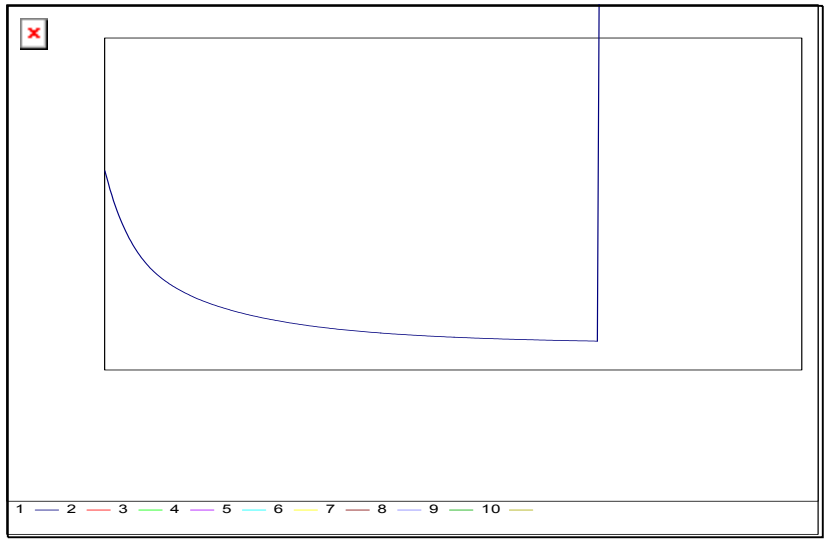

Fig. 1 – Angelica 2006 - 2007

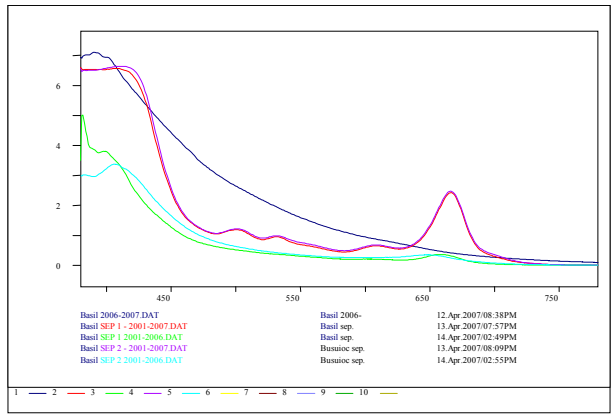

Fig. 2 - Basil

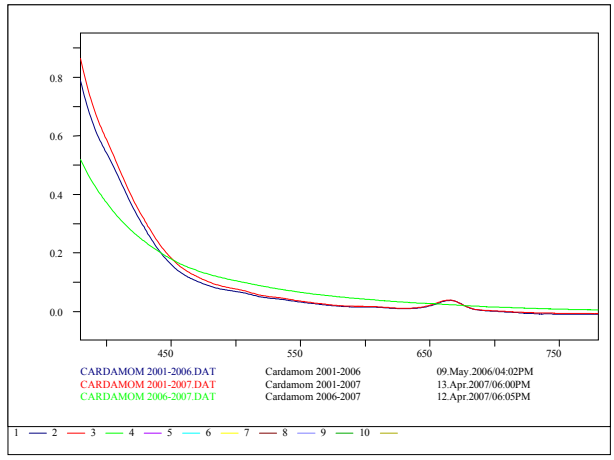

Fig. 3 - Cardamom

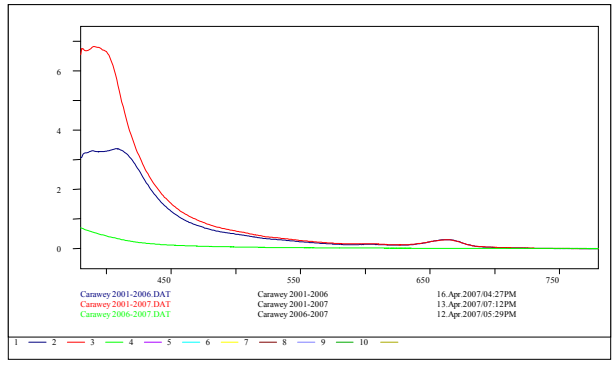

Fig. 4 - Caraway

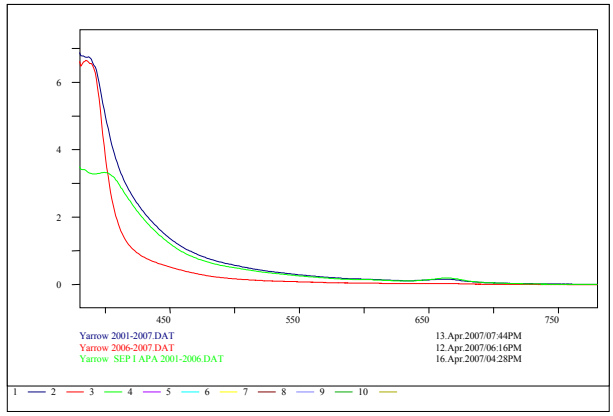

Fig. 5 - Yarrow

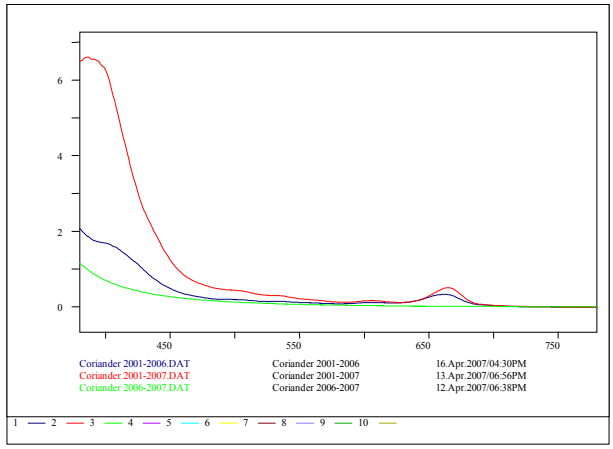

Fig. 6 - Coriander

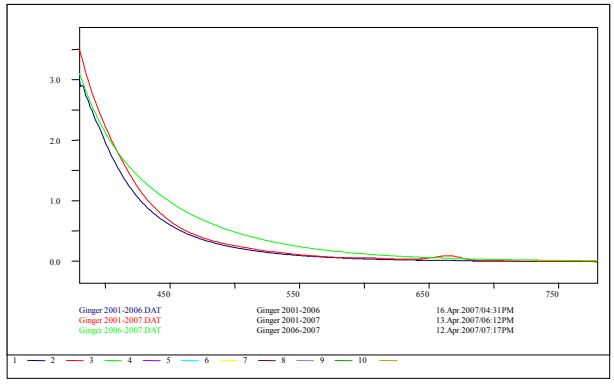

Fig. 7 - Ginger

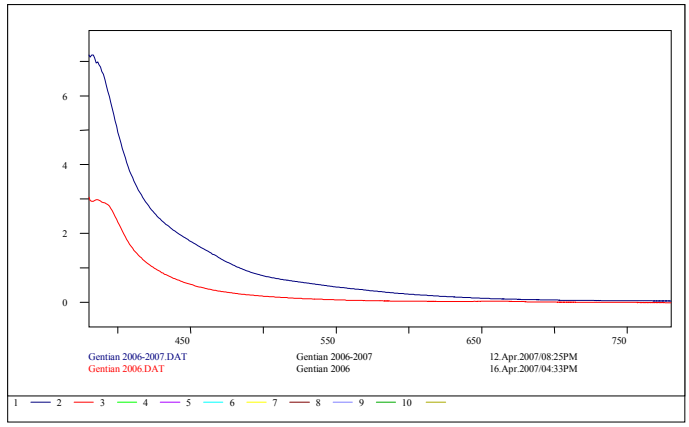

Fig. 8 - Gentian

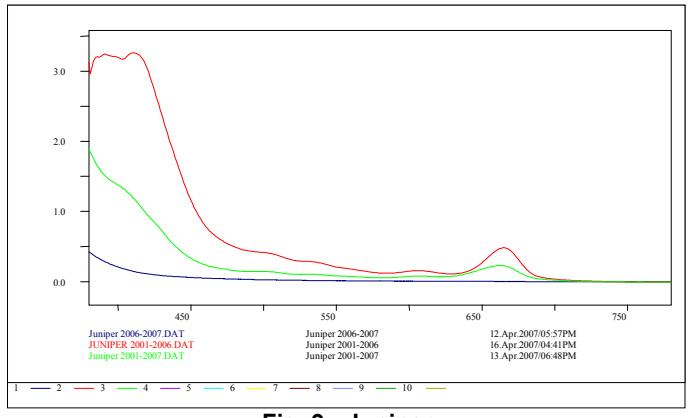

Fig. 9 - Juniper

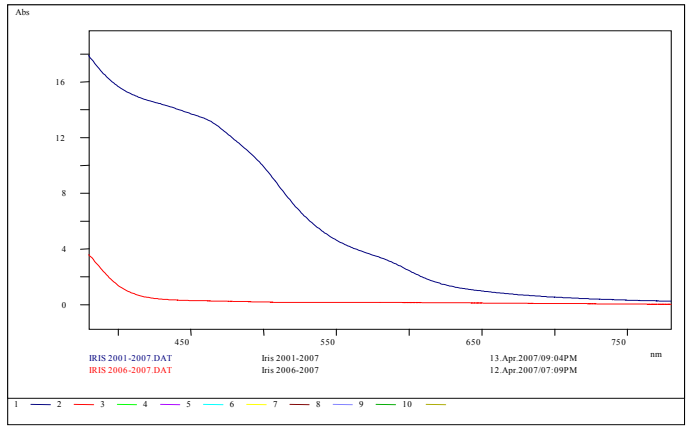

Fig. 10 - Iris

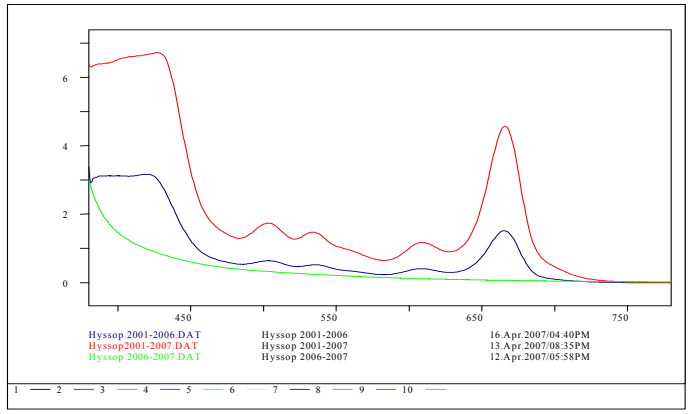

Fig. 11 - Hyssop

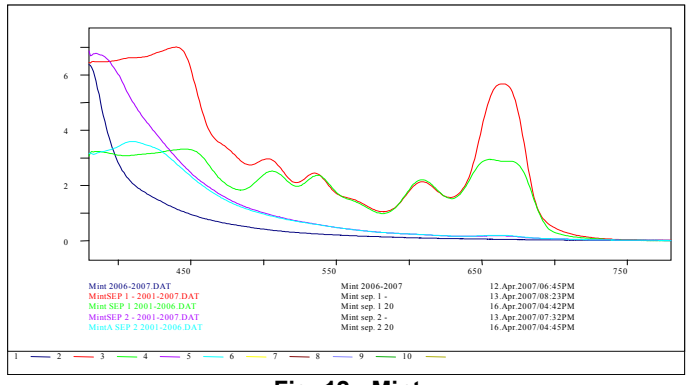

Fig. 12 - Mint

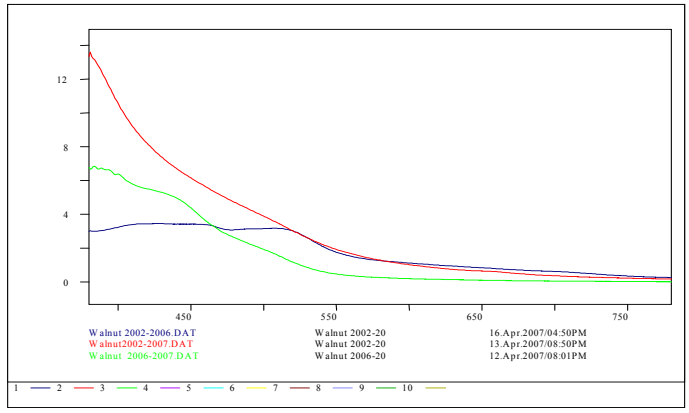

Fig. 13 - Walnut

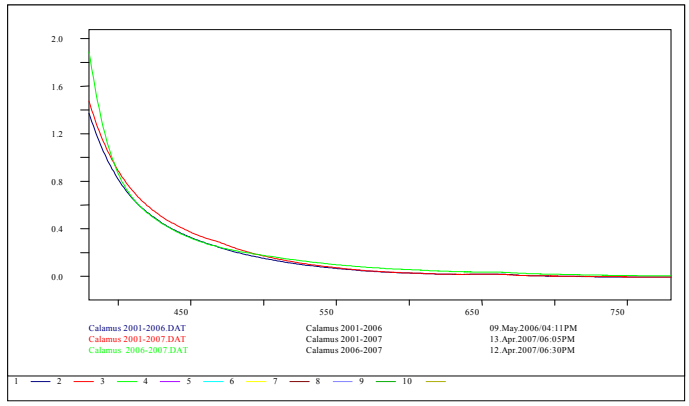

Fig. 14 - Calamus

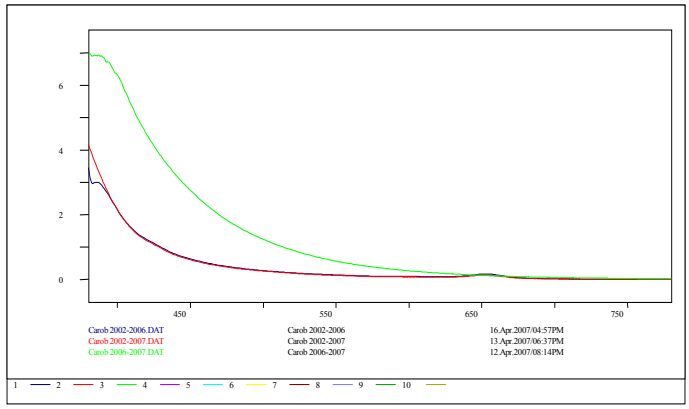

Fig. 15 - Carob

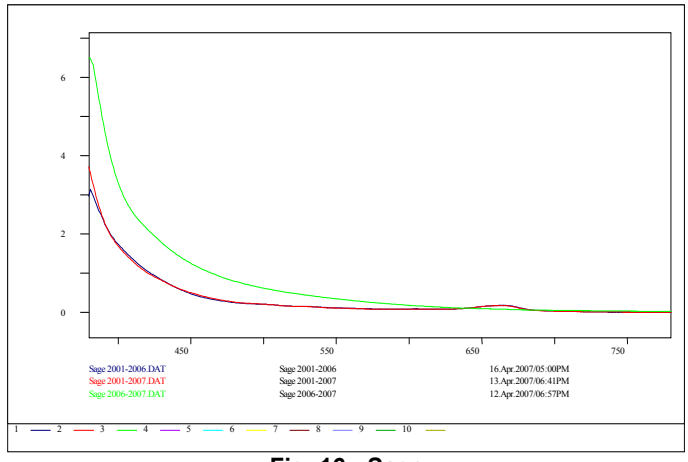

Fig. 16 - Sage

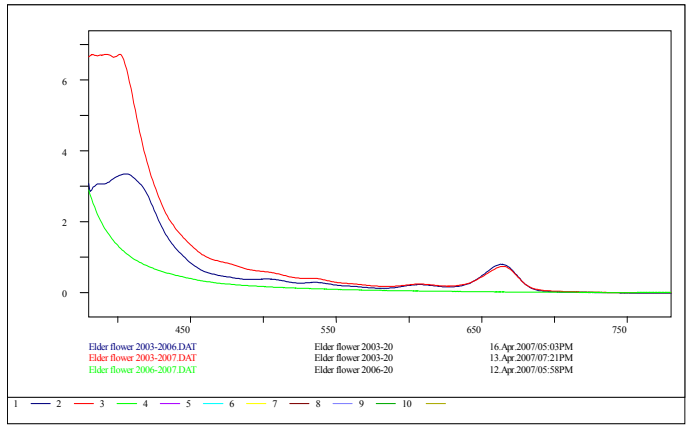

Fig. 17 - Elderflower

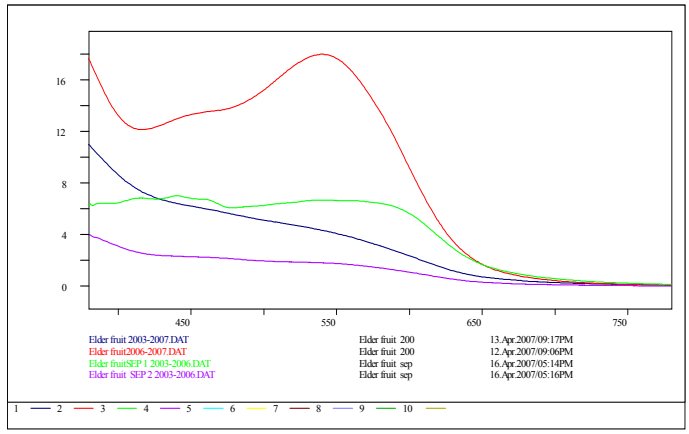

Fig. 18 - Elder fruit

CONCLUSIONS

The 18 types of hydro-alcoholic macerates of aromatic and/or medicinal plants, mostly horticultural, have shown a typical evolution, according to the assortment, from the moment of preparation and during the multiannual preservation in glass airtight containers.

A number of six macerates diminished their extract (g/100 ml), whereas in six cases, the old extracts had superior values to the fresh ones. In two cases, we did not register significant evolutions.

The content in soluble dry matter has increased in most of macerates during preservation, excepting two of them: in one case, the values remained unchanged and in another, we noticed the decrease of the content in soluble dry matter.

The macerate luminosity has evolved typically from one case to another. In eight cases, the luminosity increased (clearer macerates), in other three cases, no modifications were registered, and in seven cases, the luminosity diminished (more macerates that were opalescent).

Chromaticity had the same type of evolution, more precisely for seven samples, the colour of the old macerates was more reduced, as compared to the one existing in the fresh macerates, whereas for the remainder, we registered the contrary phenomenon.

The balance between yellow and blue was in all macerates towards yellow, though the hues were relatively different. We could notice an evolution in time, nine macerates amplifying the yellow predominance, whereas the remainder diminished it or even kept it unchanged.

The balance between red and green was present in four different situations: red predominance, green predominance, passing from the red predominance to the green one, or conversely from the green predominance to the red one.

We may consider that the determinations carried out in dynamics reflect the evolution of the chemical composition of the hydro-alcoholic concentrates, made during 5 years.

A number of 18 spectra were carried out, in order to compare the two types of macerates, the fresh ones from 2007 and the ones kept for several years (determined both in 2006 and in 2007). Determinations represent a basis of repeatability and possible identification.

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