

Effect of micella interesterification on fatty acids composition and volatile components of soybean and rapeseed oils

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RESUMEN

Efecto de la interesterificación en fase miscelar sobre la composición en ácidos grasos y componentes volátiles de aceites de soja y de colza.

Se ha llevado a cabo la interesterificación en fase miscelar de aceites de soja y de colza usando un 0.2%, 0.4% y 0.6% de níquel como catalizador, a diferentes temperaturas (60, 90 y 120°C) durante 2, 4 y 6 horas. Las condiciones de reacción de interesterificación propuestas para obtener un aceite con niveles de ácidos linolénicos bajos fueron 0.2 % de níquel a 120°C durante 4 horas, 0.4 % de níquel a 90°C durante 4 horas y 0.6 % a 60°C durante 4 horas. Se han estimado la composición en ácidos grasos y el análisis químico de los aceites interesterificados y no-esterificados. Las muestras seleccionadas se sometieron a calentamiento a 180°C durante 4 horas determinando los componentes volátiles. La aparición de algunos componentes apoyó el proceso de interesterificación por modificación de los ácidos grasos constituyentes de los aceites.

PALABRAS-CLAVE: Aceite de colza - Aceite de soja - Ácidos grasos (composición) - Componentes volátiles - Fase miscelar - Interesterificación.

SUMMARY

Effect of micella interesterification on fatty acids composition and volatile components of soybean and rapeseed oils.

Micella interesterification of soybean and rapeseed oils was carried out using 0.2, 0.4 and 0.6 percentages of nickel catalyst, each at different temperatures of 60, 90 and 120°C for 2, 4, and 6 hours. The proposed interesterification reaction conditions to obtain an oil with low linoleic acid level were 0.2 % nickel catalyst at 120°C for 4 hours, 0.4% nickel catalyst at 90°C for 4 hours and 0.6% at 60°C for 4 hours. Fatty acid composition and chemical analysis of the interesterified and non-esterified oils were estimated. Selected samples undergo heating at 180°C for 4 hours determining the volatile components. The appearance of some components supported the interesterification process for modification of fatty acid constituents of the oils.

KEY-WORDS: Fatty acids (composition) - Interesterification - Micella - Rapeseed oil - Soybean oil - Volatile components.

1. INTRODUCTION

Intesterified process is usually involves at least two different oils that have different fatty acids composition (1). Types of interesterification discussed are (a) interchange between a fat and free fatty acids, in which the most important reaction is the introduction

of acids of low molecular weight into a fat with higher fatty acids; (b) interchange between a fat and an alcohol, e.g. with glycerol, in order to produce emulsifier like monoglycerides; (c) rearrangement of fatty acid radicals in triglycerides, the so-called transesterification which in recent years has taken on the same importance as hydrogenation or fractionation. Interesterification process is often desirable to alter the composition of acyl moieties of the naturally occurring triacylglycerols properties (2-5). The classical interesterification is characterized by randomization in distribution of acyl moieties in the triacylglycerol molecules by applying a chemical catalyst such as sodium methoxide, sodium potassium alloy, metallic sodium and sodium hydroxide (5-7).

On the other hand, linolenic acid is the major precursor of flavour in soybean oil. So the increase in stability was attributed to a decrease in the amount of linolenic acid (8). The fish odour generated by use of soybean oil at frying temperature disappeared when the linolenic acid content was reduced to below 2.0% by blending with cottonseed oil (9). Autoxidation of soybean oil and the resulting formation of odour and flavour have been investigated for many years (10). Buttery flavour is known to be associated with reverted flavour in autoxidized soybean oil. The identification of volatile components of a food, the mechanisms for their formation, and / or their characteristics odour and flavours comprise the formation needed before GC analysis can be consider reliable and useful for correlation studies (11).

The aim of this work was to study the effect of interesterification process on improving flavour and frying quality of soybean and rapeseed oils.

2. MATERIALS AND METHODS

— Bleached soybean oil and Nickel catalyst (purity 91%) were supplied from Cairo Company of Oils.

— Rapeseed was supplied from Agriculture Society Giza and rape seed oil was obtained by solvent extraction in laboratory.

— Oleic acid was purchased from Chemical Company, Egypt. The purity of oleic acid was

chaked by thin layer chromatography and gave one spot.

— n-Hexane and other solvents was analytical grade.

2.1. Interesterification methods

The interesterification reaction was carried out by adding oleic acid to soybean oil with ratio 1:2 (w/v) in n-hexane and 0.2% of nickel as a catalyst at 60°C with stirring for 2 hours in an oil bath.

The same experiment was repeated for 4 hours and 6 hours respectively under the same condition of temperature and catalyst concentration. This experiment is also re-done at temperature 90°C and 120°C under the above conditions. The aforementioned experiment was repeated using 0.4% and 0.6% of nickel catalyst under the same conditions of temperature and stirring hours. The oil samples were analyzed for their iodine value (I.V), saponification value (S.V) and fatty acid composition according to method described in (12). Volatile components were evaluate for interesterified soybean oil under the condition of 0.4% nickel catalyst at 90°C for 4 hours.

Concerning interesterification of rapeseed oil just one condition was done. This condition was 0.4% nickel catalyst at 90°C for four hours.

Iodine, saponification values, fatty acid and volatile components were investigated according to the method described in (12). Oxidizability was calculated according to (13).

2.2. Extraction of rapeseed oil

Rapeseed oil were subjected to solvent extraction using redistilled n-hexane. The rapeseeds were ground, soaked in hexane for a day at room temperature. Aliquots of fresh solvent were added to the rapeseed meal to extract more oil from the seeds. The collected micella were filtered and the solvent was removed under reduced pressure to obtain the oil.

2.3. Preparation of the aroma concentration

This method was described according to (14) and carried out on the recommended samples of soybean and rapeseed oils which subjected to interesterification process using 0.4% nickel as a catalyst at 90°C for 4 hours of stirring.

2.4. Gas-liquid chromatography analysis of fatty acids and aroma concentration

The identification of the volatile components was done by using gas liquid chromatography under the following conditions:

— Column package 20% Diethylene glycerol succinate (DEGS) on chromosorb W (60-80 mesh), column length 6 feet with internal diameter 1/4 inch (stainless steel); column temperature for flavour 70 - 190°C with programming rate 4°C/ min, injection temperature 220°C, detector temperature 300°C, carrier gas (He) flow rate 30 ml / min; hydrogen flow rate 30 ml / min, air flow rate 300 ml / min.

The conditions of GLC analysis for fatty acids were as mentioned before except the temperature of column being isothermal at 195°C.

3. RESULTS AND DISCUSSION

3.1. Interesterification process of soybean oil 0.2 % nickel

It was carried out at 60, 90 and 120°C each for 2, 4 and 6 hours. The data for the fatty acid pattern of the interesterified soybean oil (table I) was demonstrated that the levels of stearic acid as a saturated fatty acid and oleic as an unsaturated fatty acid of the soybean oil were increased compared with the non interesterified oil. The concentration of linoleic and linolenic acid were decreased. The content of palmitic acid was nearly unchanged after interesterification process.

In general, it is worth to mention that the level of the certain acids were gradually increased or decreased by prolonging the stirring period. Also, interesterification process caused an increase in the percentage of total saturated fatty acids and a decrease in the percentage of total unsaturated fatty acids.

Dealing with the effect of various temperatures (60, 90 and 120°C) during interesterification of soybean oil using 0.2 % nickel and stirring for 6 hours, one would deduce the following remarks. The reason for choosing shaking period of 6 hours was to accelerate the interesterification and reach the reaction equilibrium state of ester exchange. The level of palmitic, oleic and linolenic was nearly remained constant. In other words, the rise in temperature during interesterification had no obvious effect on substitution of the aforementioned acids by the addition of oleic acid. Since the main purpose of the interesterification was to reduce the concentration of linolenic acid. The proposed interesterification reaction conditions to obtain an oil with low linolenic acid level was 120°C using 0.2% nickel and stirring period for 4 hours.

3.2. Interesterification of soybean oil using 0.4% nickel catalyst

The interesterification process was conducted for 2, 4 and 6 hours at 60, 90 and 120°C. The fatty acids

Table I
Fatty acid profiles of modified soybean oil samples produced by interesterification using nickel (0.2%) at 60, 90 and 120°C and various stirring periods

Stirring time (hr)	16:0	18:0	18:1	18:2	18:3	TS ^(a)	TU ^(b)	Oxidizability ^(c)
Control	10.9	2.3	22.2	55.7	8.9	13.2	86.8	73.9
60°C								
2	11.2	17.5	36.7	30.8	3.8	28.7	71.3	39.1
4	11.2	17.2	38.0	30.0	3.6	28.4	71.6	38.0
6	11.3	18.3	36.6	29.8	4.0	29.6	70.4	38.5
90°C								
2	11.2	15.5	44.4	25.1	3.8	26.7	73.3	33.6
4	11.1	18.4	42.3	24.5	3.7	29.5	70.5	32.7
6	11.0	18.4	40.1	25.8	4.7	29.4	70.6	34.0
120°C								
2	11.2	5.8	42.1	35.8	5.1	17.0	83.0	46.8
4	11.3	4.3	46.4	34.6	3.4	15.6	84.4	42.3
6	11.0	5.4	40.6	38.6	4.4	16.4	83.6	48.2

a and b refer to total saturated and total unsaturated fatty acids.

C: Oxidizability = [(0.02 x Oleic%) + 1 x Linoleic% + 2 x Linolenic(%)].

16:0 = Palmitic acid

18:0 = Stearic acid

18:1 = Oleic acid

18:2 = Linoleic acid

18:3 = Linolenic acid

compositions were recorded in (table II) in which the fatty acid distribution of interesterified soybean oil under the conditions 0.4% nickel at different temperatures 60, 90 and 120°C and stirring periods (2, 4 and 6 hours), demonstrated that the level of

palmitic was nearly unchanged, the levels of stearic, oleic and linoleic were increased and decreased, respectively. The resultants of interesterified soybean oil has less linoleic and linolenic acid than non-esterified soybean oil. Generally, interesterification altered the

Table II
Fatty acid patterns of modified soybean oil samples produced by interesterification using nickel (0.4%) at 60, 90 and 120°C and various stirring periods

Stirring time (hr)	16:0	18:0	18:1	18:2	18:3	TS ^(a)	TU ^(b)	Oxidizability ^(c)
Control	10.9	2.3	22.2	55.7	8.9	13.2	86.8	73.9
60°C								
2	11.2	9.9	38.0	35.1	5.8	21.1	78.9	47.5
4	11.3	11.2	38.3	35.2	4.0	22.5	77.5	44.0
6	11.2	10.3	37.2	35.5	5.8	21.5	78.5	47.8
90°C								
2	11.3	10.5	37.4	35.9	4.4	21.3	78.2	45.5
4	11.2	12.3	37.0	35.5	4.0	23.5	76.5	44.2
6	11.1	10.7	37.5	36.2	4.5	21.8	78.2	46.0
120°C								
2	11.3	10.3	39.4	33.3	5.7	21.6	78.4	45.5
4	11.2	10.2	45.1	30.1	3.4	21.4	78.6	37.8
6	11.2	10.0	42.2	32.1	4.5	21.2	78.8	41.9

a and b refer to total saturated and total unsaturated fatty acids.

C: Oxidizability = [(0.02 x Oleic%) + 1 x Linoleic% + 2 x Linolenic(%)].

16:0 = Palmitic acid

18:0 = Stearic acid

18:1 = Oleic acid

18:2 = Linoleic acid

18:3 = Linolenic acid

Table III
Fatty acid compositions of modified soybean oil samples produced by interesterification using nickel (0.6%) at 60, 90 and 120°C and various stirring periods

Stirring time (hr)	16:0	18:0	18:1	18:2	18:3	TS ^(a)	TU ^(b)	Oxidizability ^(c)
Control	10.9	2.3	22.2	55.7	8.9	13.2	86.8	73.9
60°C								
2	11.0	10.0	38.4	34.8	5.8	21.0	79.0	47.2
4	11.2	9.5	40.5	35.4	3.4	20.7	79.3	43.0
6	11.2	10.8	36.3	37.3	4.4	22.0	78.0	46.8
90°C								
2	11.2	10.0	42.4	30.1	6.3	21.2	78.8	43.6
4	11.2	9.7	45.3	29.4	4.4	20.9	79.1	39.1
6	11.1	11.7	40.4	32.0	4.8	22.8	77.2	42.4
120°C								
2	11.2	9.6	41.4	31.4	6.4	20.8	79.2	45.0
4	11.2	10.8	40.7	32.0	5.3	22.0	78.0	43.4
6	11.1	11.0	42.3	30.5	5.1	22.1	77.9	41.5

a and b refer to total saturated and total unsaturated fatty acids.

C: Oxidizability = [(0.02 x Oleic(%) + 1 x Linoleic(%) + 2 x Linolenic(%))].

16:0 = Palmitic acid

18:0 = Stearic acid

18:1 = Oleic acid

18:2 = Linoleic acid

18:3 = Linolenic acid

fatty acid profiles of the soybean oil samples used to estimate the oxidizability of various interesterified soybean oil. Hence the interesterification reaction conditions to produce soybean oil with high stability were: 0.4% nickel as catalyst, reaction temperature 90°C and shaking period 4 hours.

3.3. Interesterification of soybean oil using 0.6% nickel

The fatty acids composition of different esterified soybean oil were shown in (table III). The results illustrated that the level of palmitic acid was nearly unaffected by various conditions of interesterification compared with the palmitic acid value of non-esterified soybean oil. The contents of stearic and oleic were gradually increased by prolonged shaking period at all reaction temperature (60, 90 and 120°C). In contrast, the percentages of linoleic and linolenic were generally decreased by increasing the stirring time at each reaction time.

Concerning the production of soybean oil characterized by low linolenic acid content and hence overcome the fishy odour during frying, the reaction conditions include 0.6% nickel, reaction temperature 60°C and stirring time 4 hours would be the method to be practically applied.

To reach one of the main objectives of this work, i-e, production of modified soybean oil characterized by low linolenic acid content and can withstand oxidative rancidity for frying proposes. The following reaction condition have to be applied practically. For production of soybean oil with low linolenic acid content the conditions were 0.2% nickel, reaction temperature 120°C, stirring period 4 hours or 0.6% nickel, reaction temperature 60°C, stirring period 4 hours. To achieve the second goal (an oil with high stability), the interesterification conditions were 0.2 % nickel, reaction temperature 90°C and stirring period 4 hours or 0.6% nickel, reaction temperature 120°C and stirrig period 6 hours.

Much research has been published on the oxidative stability of soybean oil. This oil can undergo oxidation under mild conditions to develop off-flavour and even potentially harmful compounds (15-19).

Huang *et al* (20) concluded that corn and sunflower oils were more suitable for cooking or frying because they contain only small amounts of linolenate. It is well established that the fishy odour produced from soybean oil during frying is entirely depends upon linolenic acid levels. The increased level in soybean oil is accompanied by increase of the fishy odour.

Iodine values of interesterified soybean oil (with 0.2, 0.4 and 0.6% catalyst and heated at 60, 90 and 120°C for 2, 4 and 6 hours) were lower than that of the non interesterified soybean oil (table IV).

Table IV
Iodine values of non-esterified and modified soybean oil samples produced by interesterification under various conditions

Stirring period (hr)	Reaction Temperature (°C)		
	60	90	120
0.2% Nickel			
2	110.9	111.8	106.6
4	111.6	112.3	106.3
6	108.8	110.7	105.2
0.4% Nickel			
2	108.9	110.8	103.6
4	107.4	102.9	103.9
6	105.2	104.3	101.2
0.6% Nickel			
2	111.6	94.0	56.1
4	106.4	84.5	56.5
6	109.3	80.4	56.8
Control = 127.7			

This finding was in line with the fatty acids composition of modified soybean oil samples where an increase and decrease in the percentage of the saturated and unsaturated fatty acids had take place upon interesterification. The saponification values of non-interesterified and interesterified soybean oil samples were illustrated in (table V). This values is generally indicate the chain length of fatty acids. In other words, the short chain fatty acids in triglycerides gives high saponification value and vise versa with long - chain fatty acids.

Table V
Saponification values of non-esterified and modified soybean oil samples produced by interesterification under various conditions

Stirring period (hr)	Reaction Temperature (°C)		
	60	90	120
0.2% Nickel			
2	192.4	192.6	196.7
4	193.3	194.0	196.4
6	196.0	193.9	194.5
0.4% Nickel			
2	197.5	192.6	191.6
4	195.7	196.2	191.7
6	195.7	195.0	192.5
0.6% Nickel			
2	195.6	198.4	197.3
4	192.8	196.9	193.6
6	193.5	191.8	194.4
Control = 194.2			

The volatile components of interesterified soybean oil produced by 0.4% nickel as catalyst at 90°C and stirred for 4 hours was shown. Are remarkable decrease of certain aldehyde were recorded i-e, butanal, hexanal, octanal, decanal, 2-nonanal, 2,4 nonadienal and 2,4 decadienal (table VI). On the contrary, the levels of propanal and hendecanal were increased by interesterification process. For instance, the concentration of 2,5-hexadione decreased from 2.6 to 0.11, 3-ethyl hexanone from 1.9 to 0.4, 3-ethyl octanone from 16.3 to 0.49 by interesterification.

Table VI
Volatile matter (%) of non-interesterified and interesterified soybean oils heated for 4 hr at 180°C

Compounds	% Non-interesterified Soybean oil	% Interesterified Soybean oil
Propanal	0.7	1.37
Butanal	0.7	0.41
Pentanal	ND	1.23
2-Hexanol	ND	0.88
Hexanal	0.7	0.11
Methyl valerate	ND	0.14
Methyl hexanoate	ND	0.26
Heptanol	ND	0.20
Octanal	0.4	ND
1-Hexanol	ND	0.11
Nonanol	ND	1.23
Decanol	1.1	0.11
Unidentified	-	0.02
Unidentified	-	0.152
Methyl octanoate	1.1	0.056
Hendecanal	ND	0.25
1-Octanol	1.5	ND
Dodecanol	0.4	0.14
Undecanol	0.4	ND
Unidentified	-	0.31
Unidentified	0.4	0.14
2-Nonenal	1.5	0.11
2,5 Hexadione	2.6	0.11
2,4 Nonadienal	5.2	0.36
3-Ethyl hexanone	1.9	0.4
3-Ethyl heptanon	4.8	0.38
3-Ethyl octanone	1.1	0.634
3-Ethyl nonanone	16.3	0.498
Unidentified	1.9	2.34
2,4 Decadienal	20.0	ND
Unidentified	34.8	-
Unidentified	2.6	87.30

ND Refers to not detected

Alcohol generally plays a minor role in the aroma of soybean oil (21). The level of 2-hexanol was slightly increased while heptanol, 1 hexanol, dodecanol, undecanol and 1-octanol were disappeared, by interesterification. Concerning the esters, the content of methyl octanoate decreased from 1.1 to 0.1 while the concentration of methyl valerate and methyl hexanoate were increased.

The chemical structure of the volatile matter classified into 4 different group, i-e; aldehydes (saturated and unsaturated), alcohols, ketones and esters. Aldehydes constitute about 50% of the total identified volatile substances in non-interesterified soybean oil was characterized by the highest concentration of aldehyde, being 13.2, 27.9 and 1.1 times as high as that of ketones, alcohols and esters, respectively. Interesterification possessed minor changes in the levels of aldehydic compounds compared with the non-interesterified soybean oil. However, major changes had taken place for other volatile matter classes (alcohol, ketones and esters) of the interesterified soybean oil. For instance, the content of aldehydes was 1.5, 1.9 and 8.7 times as high for alcohols, ketones and esters, respectively. Since interesterification exhibited reduction of total aldehydes (12.5%), which are one of main compounds responsible for off-flavour. This finding agreed with the fatty acid pattern of the modified soybean oil by interesterification since these oil samples were characterized by low linoleic and linolenic acids.

3.4. Interesterification process of rape seed oil using 0.4% nickel heated for 4 hours at 90°C

The iodine and saponification values of the non interesterified and interesterified rapeseed oil illustrated in (table VII). It seems that interesterification of rapeseed oil in the presence of 0.4% nickel catalyst and reaction temperature of 90°C and stirred for 4 hours has no effect on the iodine and saponification values. Also the fatty acids of rapeseed oil recorded that there is no change in the content of palmitic and erucic acids and a decrease of linoleic and linolenic acids by interesterification reaction (table VIII). The flavour of crude rapeseed oil was affected by interesterification process (table IX), which demonstrated decreased of the level of aldehydes. Hexanal disappeared whilst the concentration of nonanal, decanal, hendecanal and 2,4-nonadienal decreased. On the contrary, the quantities of propanal, butanal, pentanal and 2, 4, 6-dodecatrienal increased during interesterification. The content of ketones in the interesterified oil was greater than that of crude oil. This finding was reached since the quantities of 2, 5-hexandione, 3-ethyl octanone, 3-ethyl nonanone and 3-ethyl

heptanone were increased except 3-ethyl hexanone decreased. Concerning esters, the levels of both methyl valerate and methyl octanoate were increased from 4.52 to 7.3 and from 0.01 to 2.82, respectively. The content of methyl hexanoate was decreased from 5.74 to 1.4. The analysis of the volatile compounds revealed that the levels of alcohols decreased during interesterification. Here again, the chemical structure of the volatile compounds of rape seed oil can be classified into 4 main groups, i-e, aldehydes (saturated and unsaturated), alcohols, ketones and esters. The aldehydes represented 42.25% of the identified volatiles followed by alcohols (39.9%), esters (13.35%) and ketones (1.49%). Interesterification induced very little effect on the structures of the volatile compounds of rape seed oil, the level of aldehydic compounds was 5%. Major changes in the concentration of ketones, alcohols and esters had taken place. In this respect, the levels of esters and alcohols increased whilst, the content of the ketones decreased.

These generally agreed with that the compounds responsible for the flavour of autoxidized fats are aldehydes and ketones, and great deal of work has been done to identify these compounds (22). Great many authors agreed with all these findings (23- 26).

Table VII
Iodine and saponification values of non-interesterified and interesterified of rapeseed oil using 0.4% nickel catalyst at 90°C for 4hr

	Iodine value	Saponification value
Control	99.6	166.2
Intesterified R.S.O.	73.8	185.5

Table VIII
Fatty acid profiles of modified rapeseed oil produced by interesterification using 0.4% nickel catalyst for 4hr at 90°C

Concentration of nickel %	16:0	18:0	18:1	18:2	18:3	22:1	TS	TU	Oxidizability
Control	3.0	3.8	21.9	14.4	7.1	49.9	6.8	93.3	29.0
Modified Rapeseed oil	3.0	8.5	27.0	7.1	4.5	49.9	11.5	88.5	16.6

a and b refer to total saturated and total unsaturated fatty acids.
C: Oxidizability = [(0.02 x Oleic(%)) + 1 x Linoleic(%) + 2 x Linolenic(%)].
16:0 = Palmitic acid
18:0 = Stearic acid
18:1 = Oleic acid
18:2 = Linoleic acid
18:3 = Linolenic acid

Table IX
**Volatile matter % of non-interesterified and
 interesterified rapeseed oils heated for 4 hr
 at 180°C**

Compounds	Non-interesterified Rapeseed oil	Intesterified Rapeseed oil
Propanal	ND	1.03
Butanal	ND	2.16
Pentanal	ND	6.1
2-Hexanol	1.1	3.3
Hexanol	15.95	ND
Methyl valerate	4.52	7.30
Methyl hexanoate	5.74	1.40
Heptanol	14.51	1.40
Octanal	7.66	3.1
1-Hexanol	8.31	2.89
Nonanal	5.21	2.75
Decanal	4.52	3.61
Unidentified	2.20	0.52
Unidentified	12.57	0.22
Methyl octanoate	0.01	2.82
Hendecanal	1.48	0.65
1-Octanol	0.7	ND
Dodecanol	4.24	0.212
Undecanol	1.85	1.13
Unidentified	1.88	0.47
Unidentified	2.48	0.15
2-Nonenal	ND	0.38
2,5-Hexadione	ND	0.1
2,4-Nonadienal	0.41	0.15
3-Ethyl hexanone	0.81	0.124
3-Ethyl heptanone	ND	0.17
3-Ethyl octanone	0.23	1.79
3-Ethyl nonanone	0.11	1.05
Unidentified	ND	7.62
2,4-Decadienal	ND	2.06
2,4,6-Dodecatrienal	ND	0.32
Unidentified	1.01	0.25
Unidentified	—	4.24
Unidentified	0.04	21.76
Unidentified	0.45	—
Unidentified	—	0.11
Unidentified	—	15.1
Unidentified	—	0.74

ND Refers to not detected

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Recibido: Abril 1999
 Aceptado: Septiembre 1999