Technological performances of low linolenic/high oleic rapeseed oils for food and non-food application

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Abstract

Results on the low linolenic rapeseed oils frying stability were presented at the 11th International rapeseed congress in 2003 (2000 and 2001 studies).

Additional studies were undertaken in 2003 and 2004 on rapeseed oils displaying modified fatty acid (FA) composition : low linolenic (LL) content (between 1,1 and 3%), high oleic (HO) content (76,5 %), or both low linolenic (2,5 and 2,7%) and high oleic content (75%) (LL/HO).

Six pilot plant processed samples of refined rapeseed oils were evaluated for chemical properties and involved in tests for food and non-food applications.

Six room-odor tests were carried out on four experimental oil samples used as frying oil. Check was sunflower oil. Intensity and quality of the flavour were ranked by the panellists after the first, fourth and eighth frying. The 2003 study confirmed that the effect of a higher oleic content is not significant on room-odor performances. Studies of 2003 and 2005 confirmed that the low linolenic rapeseed oil at 2,5 % ranked slightly bellow the sunflower oil (paint and fishy flavours detected at the 4th and 8th frying) meanwhile low linolenic oil at 1,1 % is equivalent and even better than sunflower oil.

Regarding the chemical alteration of the oils during frying, polar component content showed that the LL/HO rapeseed oil seemed to perform better, suggesting an effect of the total unsaturation level on this property.

Chemical and physical checks were also carried out at the laboratory for lubricant uses in 2001, 2003 and 2004 studies. Better performances for hydrolytic and oxidation resistances (equivalent to a 80 % oleic sunflower oil) were obtained with the oils LL/HO oils.

Trials have finally been conducted to check the ability of such modified FA rapeseed oils as raw material for biofuel after a trans-esterification stage. LL/HO oils appeared to improve of 3 to 5 points the cetane index (EN ISO 5165) against the industrial control meanwhile other parameters have not clearly improved, leading to conclude that such modified oils are not necessary at the present time for the biodiesel market.

These works showed that the new LL/HO rapeseed cultivars could allow to extend rapeseed oil uses in both food (frying in substitution to hydrogenated fats) and non-food applications like lubricants.

Key words : rapeseed, room-odor, linolenic acid, oleic acid, frying, oxidation stability

Introduction

When used in deep fat frying, rapeseed oil gives off an odor in the kitchen which is perceived to be unpleasant (room-odor) by the homeowner, who is accustomed to peanut or sunflower oils. France, due to its own regulation, is the only country in the world to exclude oils containing more than 2% of linolenic acid for deep-fat frying. Some previous works from France and Canada have already shown that low-linolenic rapeseed oils (LL type) had a significantly better behaviour in frying than a normal rapeseed oil ; this behavior is very close to that of a sunflower oil.

Moreover, a high linolenic acid content in rapeseed oil restricts non-food applications as lubricants in warm conditions.

Several experimental cultivars with low content of linolenic acid and high content of oleic acid (HO type) are actually proposed by the plant breeding with the objective to develop food and non-food utilizations. Two studies (2000 and 2001) led to define the minimal limit acceptable for linolenic acid in order to obtain equivalent performances in room-odor between rapeseed oils and sunflower oils. The oil with a linolenic acid content of 1.1% is equivalent to sunflower after the first, the fourth and eighth frying. In contrary, panelists judged the oils with 2.2% and 3.5% of linolenic acid to have significantly higher intensities for fishy and paints odors. A high content in oleic acid did not appear to improve the flavor quality: the oil with 2.2% of linolenic acid and 64.3% of oleic acid was equivalent to the oil with 1.9% of linolenic acid and 78.8% of oleic acid.

Further studies were undertaken in 2003 and 2004 on six rapeseed oils displaying modified fatty acid composition : low linolenic (LL) content (between 1 and 3%), high oleic (HO) content (>75 %), or both low linolenic and high oleic contents in order to evaluate their ability to be used in both food (frying in substitution to hydrogenated fats) and non-food applications like lubricants and biofuels.

Materials and methods

Crop and Processing. Five rapeseed cultivars were selected for their low linolenic content (between 1 and 3 %) and/or for their high oleic content (more than 75%) and cropped in 2003 and in 2004 by a private company and by a French public lab. The experimental seeds were crushed in the oil pilot plant Creol. Crude oils were refined by Iterg. One additional sample of refined LL/HO rapeseed oil (commercial LL/HO oil) was supplied by a seed company. The fatty acids (FA) modified rapeseed oils were compared to commercial oils: conventional sunflower and rapeseed oils.

Quality assessment. Analytical determinations were done on crude oils and refined oils ; analysis of frying oils were also done : peroxyde values according to NF T60-220, oleic acidity according to NF EN ISO 660, fatty acids analysis by gas chromatography according to NF EN ISO 5509 and NF EN ISO 5508, iron and copper by atomic absorption (NF EN ISO 8294), phosphorus by atomic absorption (IUPAC 2.423), tocopherols and tocotrienols by high performance liquid chromatography (IUPAC 2.432)(ISO 9936), polar compounds in frying oils (NF EN ISO 8420).

Frying performances.

LL and LL/HO experimental oils were evaluated for their performances in frying.

Room-odor tests. Room odor tests allow an evaluation and a comparison, in confined conditions, of flavours from two frying baths during cooking of potatoes :volume of frying bath : 2.5 litters, 360 g of French fries by bath, temperature of the bath : 180°C, eight frying called F1 to F8 for each oil.

Evaluation. Frying odors are evaluated by each member of the Iterg panel (13 people) after the first, the fourth and the eighth frying. The global quality of odor is determined by a score between 2 and 10. Odors are described through five characteristics with a scale going from weak to strong : fruity, burnt, acrid, painty and fishy.

Biolubricant performances.

The LL, LL/HO but also the HO rapeseed oils were also assessed as vegetable oil bases for lubricants.

Results for the 2001 oils are also given for a most comprehensive analisis.

Several chemical and physical tests were made by an accredited private lab to check the ability of the oils in relation to lubricant applications : cinematic viscosity (ASTM D445) at 40°c and 100°c, viscosity index, pour point (ASTM D 97), anti-wear property by the HFRR test (ISO 12156, ASTM D 147), oxidation stability at 99°C of steam turbine oils by rotating bomb (RBOT) according to ASTM D 2272, oxidation and hydrolytic stability according to Baader tests at 95°C (60 ml of oil, copper spiral, 72 hours, agitation 25 rotations per minute (rtm)), demulsibility according to ASTM D 1401 (54°C, 40 ml of oil, 40 ml water, 1500 rtm during 5 minutes).

Biofuels performances.

The LL, LL/HO, and HO rapeseed oils were finally checked at the lab scale for biofuel use.

The rapeseed oil methyl esters (RME) were synthesised at the lab scale (ITERG in 2003 and a private lab in 2004) by methanolysis path using an alkaline catalysis (transesterification reaction).

The experimental RME were fully controlled according to the European standard for automotive fuels- fatty acid methyl ester for diesel engines requirements and tests methods EN 14214. Quality of the esterification was verified as required by the standard: acid index and acidity (mg KOH/:g) NF ISO 660, peroxide index (NF T60-220), water content Karl Fisher (NF ISO 8534), methyl esters content (NF T 60-703), methanol content (NF T60-701), free, mono, di and tri glycerides (ITERG methods).

Special attention was done to the following characteristics included in the requirements list: iodine index (NF EN 14111), cold filter plugging point (CFPG, EN 116) which is a climatic dependent parameter, oxidation stability through the accelerated oxidation Rancimat test (110°c, 10g/l, 3g; NF EN 14112) and Cetane index (EN ISO 5165), which were expected to be improved with the FA modification.

Finally the cold performance of the RME was assessed by the pour point measurement (ASTM 5771).

Results and discussion

Quality of the oils

Fatty compositions. The fatty acid composition of the refined rapeseed and sunflower oils is given in the table 1.

Table 1 : Fatty acid composition (%) of the modified rapeseed oils

F.A.	Oils 2003						Oils 2004	
	LL 1	LL 2.5	LL 2.7 /HO 75.4	HO 76.4	LL2.3 /HO 72.6 commercial	Sunflower (control)	LL 2.4/HO 76.8	Sunflower (control)
16:0	3.6	4	4.3	3.9	3.6	6	3.8	6.2
18:0	1.5	1.5	1.7	1.5	1.6	3.9	2.2	4
18:1	63.6	64.1	75.4	76.4	72.6	29.4	76.8	25
18:2	26.7	23.3	12.7	7.6	15.7	58.8	11	62.2
18:3	1	2.5	2.7	6.8	2.3	0.1	2.4	0.1
20:0	0.6	0.6	0.6	0.5	0.6	0.3	0.8	0.3
20:1	1.4	1.7	1.3	1.5	1.5	0.1	1.3	0.2
22:1	<0.1	0.6	<0.1	0.2	0.6	<0.1	<0.1	0.1

Quality assessment of refined oils. The quality of experimental refined oils were in accordance with the rule of the Codex Alimentarius (tables 2 and 3). The peroxyde and oleic acidity values are largely below the Codex limits: 10 meqO2/kg for the peroxyde value and 0.3% for the oleic acidity. The refining has eliminated the prooxidant metals (iron and copper) the contents of which are also largely below the Codex limits. The degumming has eliminated phosphorus (content below detection limits, fixed at 5 ppm).

Table 2 : Quality parameters of the experimented rapeseed oils-

Oils Tests 2003	Peroxide value (meqO2/kg)	Oleic acidity (%)	Iron (mg/kg)	Copper (mg/kg)	Phosphorus (mg/kg)
LL 1	< 0.1	0.03 ± *0,01*	0,05	0,01	< 5
LL 2.5	< 0.1	0.01 ± *0,01*	0.071	0.01	< 5
LL 2.7/HO 75.4	< 0.1	0.04 ± *0,01*	0.012	0.008	< 5
HO 76.4	< 0.1	0.02 ± *0,01*	0.18 ± *0.09*	<0,005	<5
LL 2.3/HO 72.6 commercial	6.3± *2,5*	ND	ND	ND	ND
Sunflower (control)	3,6 ± *1,5*	0.02 ± *0,01*	<0.005	<0,005	< 5
Tests 2004					
LL 2.4/HO 76.8	< 0.1	0.03 ± *0,01*	0.048	0.006	< 5
Sunflower (control)			0.007	0.005	< 5

ND : non determined

Table 3 : Tocopherol content of the modified rapeseed oils (mg/kg)

oils	α		β	γ		δ	Total
Tests 2003		% of total			% of total		
LL 1	183	33.4 %	<DL	358	65.4 %	6	547 ± *82*
LL 2.5	160	31.3 %	<DL	344	67.3 %	8	511 ± *77*
LL 2.7 /HO 75.4	192	35.8 %	<DL	337	62.8 %	8	536 ± *80*
HO 76.4	200	34.8 %	< DL	368	64 %	8	575 ± *86*
LL2.3 /HO 72.6 commercial	242	35.6 %	< 2	428	62.9 %	11	680 ± *102*
Sunflower (control)	659	94.8 %	23	10	1.4 %	3	695 ± *148*
Tests 2004							
LL 2.4 /HO 76.8	234	40.2 %	3	334	57.5 %	10	581 ± *87*
Sunflower (control)	684	95 %	26	8	1.1 %	2	720± *108*

DL = detection limit

The total tocopherol content of the modified rapeseed oils is similar to the content of the conventional rapeseed refined oil (500 to 870 ppm, depending on the seed quality and on the refining) – (Table 3). The composition is not affected by the fatty acid profile modification, the γ type remaining predominant (57-65%) and the α type ranking second (30-40%); in accordance with the bibliography (α-tocopherols : 25-38%, β : 0-5 %, γ : 62-70%, δ : 0-6 %, source : Manuel des Corps Gras, Lavoisier TEC & DOC, p. 131, 1992).

Frying performances

Quality assessment of frying oils. The oleic acidity of refined oils and the content of polar components were determined after the first F1 and the eighth frying F8 (table 4). Few free fatty acids have been produced in rapeseed and sunflower oils (see the oleic acidity). After 8 frying, the polar components content is in conformity with the French regulation : less than 25% of polar components for food uses. After the first frying F1, the polar components content is in conformity with what is traditionally observed with rapeseed oils : 3 to 4% of polar components. Nevertheless after the 8th frying, the best result is got with the d ouble traits LL/HO rapeseed oil in both tests 2003 and 2004 suggesting a favourable effect of the linolenic acid content but also of the total unsaturation decreases.

Table 4 : Oleic acidity and polar components in frying oils after first (F1) and eight frying (F8) – tests 2003 and 2004

Oils	Oleic acidity (% m/m)		Polar components (%)	
	after F1	after F8	after F1	after F8
Test 2003				
LL 1	0.03 ± *0.01*	0.10 ±*0.01*	4.1 ± *1.5*	13.3 ± *1.5*
LL 2.5	0.01 ± *0.00*	0.05 ± *0.01*	3 ± *1.5*	10.9 ± *1.5*
LL 2.7/HO 75.4	0.005 ± *0.01*	0.08 ± *0.01*	3 ± *1.5*	6.8 ± *1.5*
Sunflower (control)	0.02 ± *0.01*	0.07 ± *0.01*	5,9 ± *1.5*	11.5 ± *1.5*
Test 2004				
LL 2.4/HO 76.8	0.05± *0.01*	0.08 ± *0.01*	3.2 ± *1.5*	7.6 ± *1.5*
Sunflower (control)	0.03± *0.01*	0.05 ± *0.01*	5.5 ± *1.5*	13.5 ± *1.5*

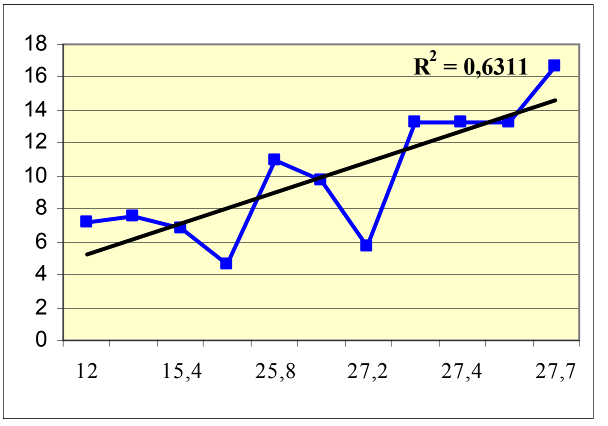

Figure 1 :Polar components (% m/m) in the modified rapeseed oils after the eight frying in relation to the C18:2+C18:3 content (%) – tests 2000, 2001,2003, 2004

NB : One value from the 2002 study (LL 2.2) was not taken into consideration

If we consider the results of all the tests run with the LL and LL/HO rapeseed oils from the year 2000, a relation ship between the polar components at F8 and the sum of 18:3 and C 18:3 contents can be established with a correlation coefficient of 0.63 (figure 1).

Room odor

The best ranking is obtained by the LL 1% oil; confirming the study of 2000 and 2001 where such fatty acid profile was shown as equivalent to sunflower (figure 2).

The addition of the high oleic trait (LL 2,7 % /HO 75,4 % in the 2003 tests and LL 2,4% /HO 76,8 % in the 2004 test) did not lead to improved flavour performances; confirming the major role played by the linolenic content on the frying oil flavour.

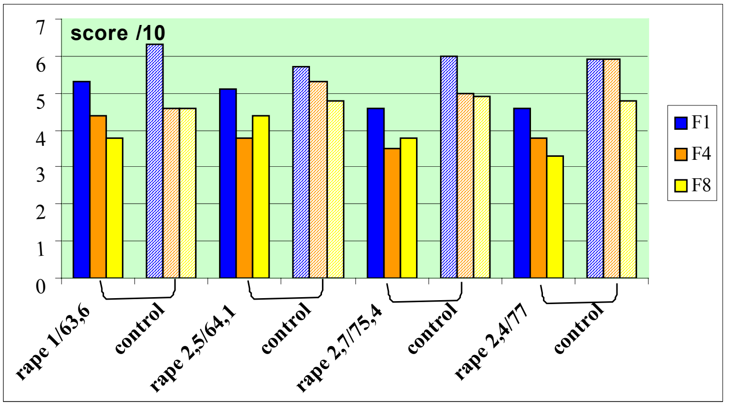

Figure 2 : Global quality assessment of the flavour after the first F1, fourth F4 and eighth frying F8 of the modified rapeseed oils tested in 2003 and 2004 in comparison to the conventional sunflower oil (control)

The results of the Student test for the four flavour descriptors and for the global quality assessment of the flavour at F1, F4 and F8 showed that the modified oils displaying 2,5% and 2,7 % of linolenic acid expressed some unfavourable flavours, especially painting fishy tastes for the LL 2,7 %/HO 75,4%, at the successive frying runs (table 5 and table 6).

These results confirm our previous study where the 2.5% C18:3 value was pointed as a kind of threshold over which some flavour differences can be detected in comparison to the sunflower oil.

Table 5 : Statistical results (Student test) of the room-odor tests 2003

Tested couple : Rapeseed oil against control (sunflower)	LL 1			LL 2.5			LL 2.7 /HO 75.4		
	F1	F4	F8	F1	F4	F8	F1	F4	F8
Global quality	1%	ns	ns	ns	1%	ns	1%	1%	ns
Lost of fruity	ns	ns	ns	ns	5%	ns	ns	1%	ns
Bean	ns	ns	ns	ns	ns	ns	ns	ns	ns
Burnt, acrid	ns	ns	ns	5%	ns	ns	ns	ns	ns
Painty, fishy	ns	ns	ns	ns	5%	ns	5%	1%	5%

ns : non significant

Table 6 : Statistical results (student test) of the room-odor test 2004

Tested couple : Rape oil against control (sunflower)	LL 2.7/HO 75.4		
Descriptor	F1	F4	F8
Global quality	5%	1%	1%
Lost of fruity	1%	5%	ns
Bean	ns	5%	ns
Burnt, acrid	ns	ns	ns
Painty, fishy	5%	1%	5%

ns : non significant

Nevertheless, it has to point that the differences were slight, that in this kind of design the score is highly related to the control oil which the rapeseed oil is compared to and that the people of Iterg involved in our room odor evaluation were hardly trained and sensitive. Additional consumers tests would be necessary to check the linolenic content which would be widely accepted for home frying use in France.

Lubricant performances

Physical and chemical characteristics.

The rapeseed oils experimented from 2001 till 2004 expressed a good viscosity index (in relation with a good lubricity) and a high flash point (reducing fire risks), in accordance with the fluid vegetable oils properties (table 7). The pour point was stable between years (-21 till -27°c) and was slightly better than the controls, however as for most vegetable oils, an additive should be necessary, depending on the conditions of use. (table 7, table 8).

The Noack volatility was very low and similar to the controls, in accordance with the behaviour of the fluid vegetable oils and their ability to decrease the lubricant dispersion in the atmosphere (good for the health impact on the operators) (table 7, table 8).

Table 7 : Physical and chemical properties of the rapeseed oils – tests 2001, 2003 and 2004

	Oils 2001			Oils 2003					Oil 2004
	LL 1.1	LL 1.9 HO 78.8	HO 77.1	LL 1	LL 2.5	LL 2.7/ HO 75.4	HO 76.4	LL 2.3 /HO 72.6 commercial	LL 2.4 HO 76.8
Viscosity at 40°c (mm^2/s)	35.91	38.64	36.65	35.78	33.38	37.49	36.73	37.38	37.89
Viscosity at 100°c (mm^2/s)	7.88	8.32	8.21	8.17	8.1	8.3	8.27	8.34	8.37
Viscosity index	200	199	209	214	230	210	211	209	206
Pour point (°c)	-27	-21	-21	ND	-23	-23	-22	-23	-20
Flash point (c°)	200	239	187	ND	ND	ND	ND	ND	ND
Noack volatility (%)	0.34	0.25	0.51	ND	ND	ND	ND	ND	ND

ND : non determined

Table 8 : Physical and chemical properties of the controls: commercial conventional rapeseed oil and sunflower oils (without additives)

	Conv rapeseed	**HO 80 sunflower**	**HO 90 sunflower**
Viscosity at 40°c (mm^2/s)	39.8	39.9	38.8
Viscosity at 100°c (mm^2/s)	8.9	8.7	9
Viscosity index	213	205	224
Pour point (°c)	-18	-12	-18
Flash point (°c)	269	265	272
Noack volatility (%)	0.4	0.37	0.3

Lubricants tests

The first tests of 2001 had highlighted a good anti-wear property of all the rapeseed oils whatever the LL and HO modification, in accordance with the literature references available for fluid vegetable oils. That's why we did not replicate such tests in the following years (Table 9).

The modified rapeseed oils expressed also acceptable water separation properties (table 9) with average times of 15-20 minutes before obtaining the separation of the oil base from the water.

Regarding both oxidation tests (RBOT at 40°c and Baader test), the LL modification did not lead to a clear improvement of the performances compared to the commercial conventional rapeseed oil. (Table 9, figures 3,4)

The best results, equivalent to the 80% oleic sunflower oil, were obtained by experimental oils combining LL and HO traits, suggesting a favourable effect of the total unsaturation decrease and of the linolenic acid lowering as for the oil frying oil stability (figures 3,4).

However none of the evaluated rapeseed oils reached the performances of the 90% oleic sunflower oil, suggesting that the hydrolytic and oxidation stabilities of such modified rapeseed oils remain insufficient for the most sensitive lubricant uses (some hydrolytic and all the motor oils lubricants)- figures 3 and 4

Table 9: Physical and chemical properties of the modified rapeseed oils

	Oils 2001			Oils 2003					Oil 2004
	LL 1.1	LL 1.9/ HO 78.8	HO 77,1	LL 1	LL 2.5	LL 2.7/ HO 75.4	HO 76.4	LL 2.3/ HO 72.6 com.	LL 2.4/ HO 76.8
Wear test HFRR (mm)	0.68	0,.	0.69	ND	ND	ND	ND	ND	ND
Demulsibility ml oil/ml water/ml emulsion/(min)	43/36/1 (20)	42/37/0 (15)	43/37/0 (15)	40/40/0/15	40/40/0 /15	40/40/0/ 15	41/37/2/ 15	40/38/2/ 25	41/37/2/20
Oxidation stability RBOT at 99°C(min)	64	125	90	91	97	99	100	97	130
Oxidation stability Baader, variation of TAN (mg KOH/g)	0.15	0.24	0.38	0.001	0.475	0.34	0.60	0.5	0.26
Oxidation stability Baader, variation of viscosity at 40°C	29.29	20.52	28.05	39.55	49.7	25.85	22.6	36.44	25.73

ND : non determined

Table 10 : Physical and chemical properties of the controls.

	Conv rapeseed	HO 80 sunflower	HO 90 sunflower
Demulsibility ml oil/ml water/ml emulsion/(min)	30/40/1 (20)	40/40/0 (20)	40/40/0 (20)
Oxidation stability RBOT at 99°C (min)	61	130	>250
Oxidation stability Baader, variation of TAN (mg KOH/g)	0.43	0,3	0
Oxidation stability Baader, variation of viscosity at 40°C	29.5	20	14.2

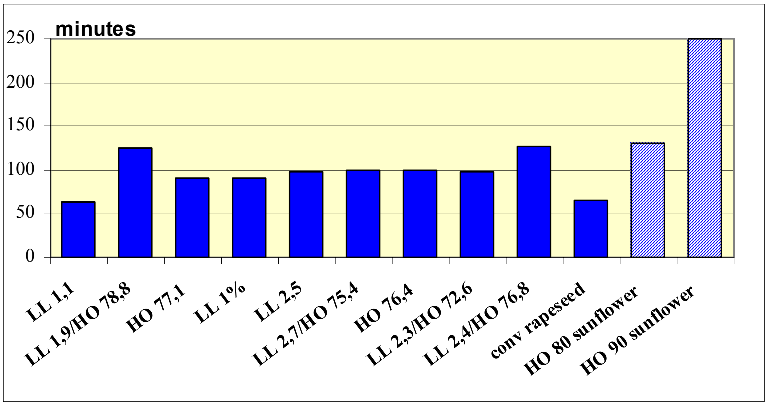

Figure 3 : Oxidation stability, RBOT test at 99°c, time to obtain a pressure depression of 175 KPa, tests 2001, 2003 and 2004.

Biofuels performances

All the methyl esters obtained from the modified rapeseed oils fitted a good quality.

The iodine index of the LL or HO RME is improved compared to the control and is fare bellow the maximum limit of 120 included in the FAME biofuel standard CEN 14214.

The LL or HO RME delivered good cold performances (pour point and CFPP) similar to the controls.

The cetane index of the FA modified rapeseed oils was found to also meet the European standard CEN 14214 (>51) and to be improved with the decrease of LL content and/or the increase of the HO content. In the 2003 tests, the double modified type (LL/HO) surpassed the other modified rapeseed RME for the cetane index.

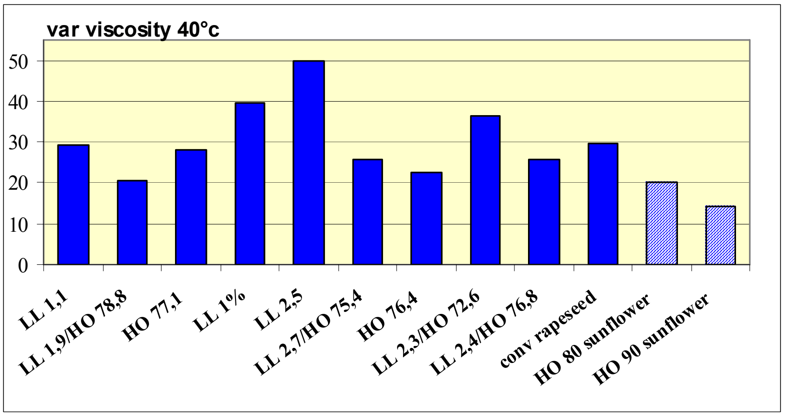

Figure 4 : Oxidation stability in the Baader test at 95°c; variation of the viscosity measured at 40°C before and after the test in the 2002, 2003 and 2004 experiments.

First reliable results in the Rancimat test were obtained in 2004 suggesting a better oxidation stability of the LL/HO rapeseed oil type compared to the conventional one. Further controls would be necessary to quantify the difference.

All in all the LL/HO modification of the rapeseed oil could be a way of biofuel quality improvement.

Table 11 : Physical and chemical characteristics of the modified rapeseed methyl esters (RME)

	RME 2003					RME 2004	
oils	LL2.5	LL2.7/ HO 75.4	HO 76.4	LL 2.3/ HO 72.6 commercial	Control Commercial RME	LL 2.4/ HO 76.8	Control commercial RME
Iodine value	111	99	94	ND	ND	96 ± *3*	117 ± *3*
Peroxide index (meq O2/kg)	9	3.3	3.95	1.5 ± *2*	5 ± *2*	2.5± *1*	2± *1*
CFPP (1) (°c)	-17	-15	-19	- 16	- 10	-10	- 13
Pour point (°c)	- 12	- 10	- 13	- 21	- 15	-11	- 12
Trouble point (°c)	- 4	- 4	- 5	- 2	- 3	- 2	- 7
Cetane index	58.3	59	56.75	53.3	50.1	53.9	51.8

(1) : cold filter plugging point
ND : non determined

Table 12 Oxidation stability of the modified rapeseed oil evaluated in 2004

	RME 2004	
	LL 2.4/HO 76.8	Control commercial RME
Oxidation stability (Rancimat), induction time in hours	18.5 ± *2.8*	8.1 ± *1.5*

NB : Commercial RME is not additivated

Conclusion

The new low linolenic and high oleic rapeseed cultivars provided in Europe by the breeding activity confirmed improved performances of the oil in hot conditions compared to the conventional FA profile. Regarding frying behaviour, the quality flavour was confirmed to be acceptable until 2 to 3 % of linolenic acid in the oil in our experiments and was not dependent on the oleic content. These results would have to be confirmed in consumer tests.

The association of high oleic (> 75%) and low linolenic (< 2.7 %) traits gave the best technological performances of the oil, especially for oxidation stability in both food and non-food evaluation. Therefore we can expect for the LL/HO rapeseed oils new markets in the frying food as well as in the lubricant fields. Such cultivars will also offer a way to enhance the quality of the biofuel while being processing as methyl esters.

To conclude may we imagine that in a near future the low linolenic/high oleic rapeseed varieties will become the commodity type meanwhile the conventional profile (with 7-10 % of linolenic acid) will move to a speciality type for their nutritional value ?

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