

EFFECT OF HUMID AND DRY HEAT TREATMENTS ON THE QUALITY
OF THE OIL AND CAKE OF RAPESEED
"BRASSICA NAPUS"

BY

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ABSTRACT

Dry heat (roasting) and humid heat (autoclaving) treatments had led to an appreciable changes in some physical and chemical properties of rapeseed meal and oil. Also, caused some changes in total and the individual percentages of saturated and unsaturated fatty acids. Autoclaving treatment showed a remarkable reduction in the amount of erucic acid (C_{22:1}), reached to 68.77%.

Fractionation of hydrocarbon and sterol compounds of the treated oil by G.L.C. technique showed that the C₂₄ was the major component in the hydrocarbon fractions. B-sitosterol, was the main sterol component in sterols, and showed a remarkable increase during heat treatments.

Humid treatments showed a little effect on the total glucosinolates and the activity of myrosinase enzyme of rapeseed. On the contrary, dry heat treatments had certainly minimized the amount of total glucosinolates and inhibited to a great extent the myrosinenzyme activity.

INTRODUCTION

German varieties of low erucic acid rapeseed has been introduced to Egypt as a cheap source for edible oil consumption. However, the oil and cake of rapeseed might contain some other toxic undesirable materials.

Hung (1977) and Sauer & Kramer (1980), confirmed that rats fed with high erucic acid rapeseed oil showed evidence of myocardial lipidosis. The latter author stated that this acid might lead to inactivation of the oxidative enzymes shared in B-oxidation.

Kramer *et al.* (1983), mentioned that erucic acid (C_{22:1}) is poorly metabolized and consequently fats could be accumulated in the heart muscle, adrenal gland and ovarian tissues. Kramer (1987), mentioned that the toxicity of rapeseed oil might be also attributed to the hard imbalance of the nutritional fatty acids in this oil.

El-Nockrashy *et al.*, (1977) and Langer & Greer (1977), stated that even rapeseed protein contains high concentrations of its glucosinolate contents which by myrosinase enzyme were harmful. It is responsible for the enlargement of animal thyroids fed on diet containing high level of rapeseed cake.

Josefsson (1975), studied the effect of heat treatment on rapeseed meal and myrosinase enzyme in relation with time and moisture. Rauchberger *et al.* (1979), studied the effect of heat treatment of glucosinolate on the nutritional quality of rapeseed meal.

The aim of this study was twofold; firstly; to study the effect of heat treatment on both oil and meal constituents in a trial to improve their quality, secondly; to inactivate the myrosinase enzyme for minimizing the toxic products resulting from glucosinolate decomposition.

MATERIALS AND METHODS

1- Materials:

Rapeseed (*Brassica napus*), variety Lesira 145 samples were obtained from Agricultural Research Centre, Oil Crops Dept., Giza.

Mustard seeds (Black) were obtained from Faculty of Medicine, Giza. They were used for the preparation of myrosinase enzyme to determine the glucosinolate compounds.

Pure D-glucose analar was purchased from (B.D.H.) Co.

2- Standards:

a- Standard fatty acids of C₁₀, C₁₂, C₁₄, C₁₆, C_{16:1}, C₁₈, C_{18:1}, C_{18:2}, C_{18:3}, C₂₀, C_{22:1}, A.R. grade (>99% by G.L.C.) were obtained from Koch Light Laboratories LTD; England.

b- Standard unsaponifiable matter hydrocarbon C₂₀, C₂₂, C₂₈, C₃₀, and C₃₂, squalene, α -tocopherol, cholesterol,

campesterol, B-sitosterol, A.R. grade (>99% by G.L.C.) were obtained from Koch Licht Laboratories, LTD; England.

- c- Standard curve of pure D-glucose was carried out to determine total soluble carbohydrates using the method reported by Helbert and Brown (1957).
- d- Standard curve for determining total glucosinolates compound as reported by Hultman (1959).

3- Heat treatments of rapeseeds:

Rapeseeds were autoclaved (heating with high pressure, 120°C, 1.5 p.s.i.) for 5, 10 and 20 min. The seeds were air-dried, then grounded and kept until use.

Rapeseeds were roasted (dry heating) at 100, 120 and 140°C for 30 and 60 min., respectively; then the seeds were grounded and kept until use.

The oil was extracted from the grounded seeds by using n-hexane according to the method mentioned by Rady et al., (1987).

4- Analysis of crude oil:

The refractive index, specific gravity, acid value, peroxide value, iodine value, saponification value and the amount of the unsaponifiable matter were determined in the different oil samples obtained from different treatments as reported by A.O.A.C. (1980).

5- Determination of fatty acids and unsaponifiable matter:

The methyl esters of the fatty acids from different oil samples were prepared using sulphuric acid in methanol 2.5% as a methylated agent. The methylation process was carried out as reported by Anon (1966) and the methylated fatty acids samples were subjected to G.L.C. analysis using Pye-Unicam instrument (model 104). The nitrogen, hydrogen and air flow rates were 60, 60 and 30 ml./min.; respectively, the chart speed was one cm/min.; attenuation was 50×10^{-2} , column temperature of 200°C and detector temperature of 250°C were used. Standard methyl esters of the above-mentioned fatty acids were used as standard authentic samples. The percentage of each individual fatty acid was determined according to Nelson et al., (1969).

The unsaponifiable matter of different samples were also determined by G.L.C. technique. The flow rates of different gases were: nitrogen 30 ml/min.; hydrogen 33

ml/min. and air 330 ml/min. The chart speed was 2 cm./min. and the attenuation was 32×10^{-2} . Quantitative identification was carried out on the basis of peak area measurements as reported by McNair and Bonelli (1969).

6- Analysis of meal:

Moisture, ash, oil, protein and crude fibre contents of the different meal samples were determined according to A.O.A.C. (1980).

The total soluble carbohydrates were estimated according to the method described by Helbert and Brown (1959).

The determination of cations was carried out according to the method described by Rwoe (1973), by using a Pye Unicam atomic absorption spectrophotometer, Sp 1900.

Estimation of glucosinolates was carried out by the liberated glucose during hydrolysis of the glucosinolates by myosinase enzyme (Rauchberger *et al.*, 1979).

RESULTS AND DISCUSSION

1- Effect of humid and dry heat treatments on the physical and chemical properties of rapeseed oil:

The results in table (1) illustrated the chemical and physical characteristics of rapeseed oil and the effect of autoclaving and roasting treatments on these properties. These results indicated that physical properties i.e. refractive index, specific gravity and also some chemical properties i.e. saponification, acid values and unsaponifiable matter percentage in untreated sample were in agreement with that reported by Khalil, (1978); Afifi (1985) and Farag *et al.*, (1986) for cotton seed, rice bran and rapeseed oils, respectively. While, iodine value was less than that stated by the above-mentioned investigators, this observation might be attributed to that oil contained less amounts of linoleic ($C_{22:2}$) acid, table (2).

The obtained results showed that, autoclaving and roasting heat treatments had almost no effect on the refractive index of rapeseed oil. Similar results were reported by Rady *et al.*, (1987) on soybean oil. Also, both treatments had very minor effect on the gravity of rapeseed oil. Autoclaving however had led to a noticeable decrease in acid values of rapeseed oil. These results are in agreement with the results reported by Rady *et al.*, (1987).

Table (1) : Effect of humid and dry heat treatments on physical and chemical properties of rapeseed oil .

C o n t e n t s	C o n t r o l	H e a t t r e a t m e n t s						A u t o c l a v i n g a t 1 2 0 ° C / 1 . 5 p . s . l .		
		1 0 0 ° C		1 2 0 ° C		1 4 0 ° C		5 m i n .	1 0 m i n .	2 0 m i n .
		3 0 m i n .	6 0 m i n .	3 0 m i n .	6 0 m i n .	3 0 m i n .	6 0 m i n .			
Refractive index/25°C	1.4693	1.4702	1.4698	1.4702	1.4703	1.4707	1.4715	1.4696	1.4688	1.4698
Specific gravity/25°C	0.9115	0.9099	0.9093	0.9077	0.9102	0.9107	0.9118	0.9096	0.9047	0.9046
Acid value	0.72	0.72	0.69	0.70	0.70	0.69	0.69	0.58	0.61	0.56
Peroxide value (meq/kg)	3.33	3.92	4.13	4.45	5.78	5.72	5.78	9.38	13.20	14.03
Iodine value	106.95	105.65	104.70	104.80	104.10	104.30	101.70	94.10	93.93	93.69
Saponification value	190.36	192.65	195.06	193.24	191.90	192.10	193.16	192.31	189.68	189.33
Unsaponifiable matter percentage	1.70	1.73	1.65	1.68	1.80	1.70	1.66	1.81	1.79	1.83

Humid heat treatment had induced a gradual increase in the peroxide value of rapeseed oil with prolonging autoclaving time as shown in table (1). Roasting treatments increased the peroxide values from 3.33 in control sample to 3.92, 4.13, 4.45, 5.78, 5.72 and 5.78 for samples heated at 100, 120 and 140°C for 30 min and 60 min., respectively. In other words these treatments increased the peroxide values with reference to control sample by 17.7% and 24% for samples heated at 100°C; 33.6% and 73.6% when heated at 120°C; 71.8% and 73.6% when heated at 140°C for 30 and 60 min.

It seems that heat treatments had accelerated the autooxidation and formation of peroxide values of this oil. Similar results were reported by El-Sharkawy *et al.*, (1986). Also, the obtained results indicated that dry heat treatment caused a slight decrease in iodine value i.e. from 106.95 (control sample) to 101.70 (4.9%) while humid treatment caused a noticeable decrease i.e. from 106.70 (4.9%) to 93.69 (12.40%). This decrease in iodine value might be attributed to either saturation or isomerization of unsaturated fatty acids specially at high temperatures (Rady *et al.*, 1987). Furthermore, both roasting and autoclaving had a slight effect on the total amount of the unsaponifiable matter and the saponification as reported in table (1).

2- Effect of heat treatments on fatty acids composition of rapeseed oil:

Results in table (2) show the differences in fatty acids constituents of rapeseed oil, and the effect of autoclaving and roasting treatments on it. Common edible oils e.g. cotton seed oil, soybean, sunflower and corn oils contained a high percentage of C_{18:2}, i.e. linoleic acid which ranged from 41.8% in soybean oil (Afifi, 1985) to 71.5% in sunflower oil (Khalil, 1978), this amount was greatly decreased in rapeseed oil (10.29% in control sample). On the other hand, the amount of oleic was found to be high in rapeseed variety i.e. 76.52% on the account of C_{18:2} acid. Such results simply means that this oil contains comparatively low amounts of essential fatty acid i.e. linoleic acid. Farag *et al.* (1986), stated that there are some sort of abnormalities in the fatty acid contents of rapeseed oils which made it little different from the other semidrying oils.

Data in table (2) revealed that both autoclaving and roasting treatments caused a slight changes in the total unsaturated and saturated fatty acids. Also the percentages

Table (2) : Relative amounts of fatty acids composition obtained by G.L.C. analysis in rapeseed oil subjected to different heat treatments .

Fatty acids	R.R.T.	Control sample	Autoclaving				Dry heat treatments					
			5 min.	10 min.	20 min.	100°C	100°C	120°C	120°C	140°C	140°C	
						30 min.	60 min.	30 min.	60 min.	30 min.	60 min.	
Undecanoic	0.08	0.83	0.07	0.07	-	T	T	T	T	-	T	T
Palmitic	0.47	3.71	4.33	3.80	5.56	T	4.41	4.04	4.09	4.42	T	3.58
Palmitoleic	0.54	-	-	T	T	T	T	T	T	T	T	T
Stearic	0.90	0.27	0.41	0.27	0.49	0.42	0.32	0.31	0.25	0.49	0.31	0.31
Oleic	1.00	76.52	81.26	80.58	72.64	79.97	77.92	80.03	79.37	79.39	79.53	79.53
Linoleic	1.20	10.29	8.83	9.28	13.79	10.11	10.76	9.11	9.59	9.51	9.97	9.97
Linolenic	1.50	6.13	3.61	4.11	6.02	4.32	4.72	4.61	4.77	4.38	4.59	4.59
Arachidic	1.70	1.42	1.02	1.26	1.24	1.10	1.32	1.37	1.38	1.27	1.47	1.47
Erucic	4.30	0.83	0.48	0.64	0.26	0.56	0.55	0.52	0.55	0.54	0.55	0.55
Total unsaturated fatty acids		93.77	94.18	94.61	92.71	94.96	93.95	94.27	94.28	93.82	94.64	94.64
Total saturated fatty acids		6.23	5.83	5.402	7.29	5.05	6.05	5.72	5.72	6.18	5.36	5.36

* T = Traces.

of the individual fatty acids of rapeseed oil slightly differed after heat treatments. Similar results were obtained by El-Sharkawy *et al.*, (1986) and Rady *et al.*, (1986). Humid heat treatments decreased the undesirable erucic acid content more than that in roasted seeds, i.e. erucic acid after autoclaving was reduced from 0.83% to 0.26% in the seeds after 20 min., leading to a total reduction of 68.67% in this undesirable material.

3- Effect of heat treatments on the unsaponifiable matter components of rapeseed oil:

The results obtained from G.L.C. analysis after autoclaving at 120°C/1.5 p.s.i. for 20 min. and roasting for 60 min. at 120°C were presented in table (3). The results of control sample were in a way similar to those reported by Farag *et al.*, (1986). Humid heat treatment in general had led to some decrease in the total amounts of hydrocarbons and an increase in the total amounts of sterols, table (3). On the other hand, some compounds were completely disappeared as the unsaturated C₃₂ hydrocarbon. Other new compounds were detected only after autoclaving as C₂₂ and some other unknown hydrocarbons. Such compounds might be originated from the hydrolysis of higher hydrocarbons or sterols owing to the temperature used.

Campesterol, stigmasterol and B-sitosterol were generally increased as a result of autoclaving and roasting treatments, specially, B-sitsterol which represents the main sterol compound. The increment in this material ranged from 8.19% to 14.43% and 13.18% after autoclaving and roasting seeds, respectively. Cholesterol compound was only identified in very little amounts 0.59% and 0.47% in autoclaved and roasted seeds, respectively, table (3). However, this material was not detected in the control sample.

4- Effect of heat treatments on chemical composition of rapeseed meal:

Autoclaving of rapeseed had no effect on moisture content while roasting caused a gradual decrease owing to moisture loss by evaporation, table (4). Humid and roasting treatments slightly decreased the crude fibre as shown in table (4), this decrease might be attributed to partial degradation of crude fibre by both humid and roasting treatments.

The obtained results indicated that a noticeable increase in reducing and non-reducing sugars were pointed out after roasting treatment, which might be attributed to the hydrolysis of polysaccharides to simple sugars.

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Table (3) : Relative amounts of unsaponifiable matter obtained by G.L.C. analysis in rapeseed oil subjected to different heat treatments .

Component	R.R.T.	Control	Autoclaving	Roasting at 120°C		
				30 min.	60 min.	
1 Decane	C ₁₀	0.03	-	-	11.21	2.66
2 Dodecane	C ₁₂	0.11	0.54	0.38	12.54	0.15
3 Unknown	1	0.13	0.77	0.75	-	-
4 Unknown	2	0.14	-	0.14	0.16	0.17
5 Tetradecane	C ₁₄	0.15	0.86	0.61	0.30	0.25
6 Unknown	3	0.17	-	-	-	1.19
7 Unknown	4	0.19	-	0.34	-	-
8 Unknown	5	0.20	-	0.68	0.21	T
9 Unknown	6	0.22	-	1.13	0.44	0.24
10 Hexadecane	C ₁₆	0.25	2.79	0.99	0.70	0.09
11 Unknown	7	0.26	-	-	0.57	0.47
12 Unknown	8	0.29	-	0.43	0.89	T
13 Octadecane	C ₁₈	0.33	1.63	0.54	0.29	T
14 Eicosane	C ₂₀	0.41	1.34	3.41	1.22	2.17
15 Unknown	9	0.43	-	0.41	0.18	0.17
16 n-docosane	C ₂₂	0.45	-	1.69	0.18	0.40
17 n-tetracosane	C ₂₄	0.48	46.48	39.81	44.02	55.34
18 n-pentacosane	C ₂₅	0.51	0.45	0.16	0.07	0.34
19 Unknown	10	0.59	0.59	1.00	1.70	1.12
20 n-hexacosane	C ₂₆	0.55	2.56	3.82	-	2.26
21 n-heptacosane	C ₂₇	0.56	-	0.17	-	-
22 n-octacosane	C ₂₈	0.60	1.19	1.08	1.04	1.20
23 Unknown	11	0.61	-	0.18	-	-
24 Unknown	12	0.64	-	5.25	-	2.91
25 n-nonacosane	C ₂₉	0.66	0.45	0.38	0.17	-
26 Squalene		0.68	0.62	0.26	0.29	0.21
27 n-tritricontane	C ₃₀	0.70	1.13	1.15	0.07	0.18
28 n-untriacontane	C ₃₁	0.72	2.23	2.69	0.11	0.63
29 Unknown	13	0.74	5.47	1.84	0.33	2.03
30 n-dotriacontane	C ₃₂	0.76	3.74	-	0.21	1.00
31 n-dotriacontane	C ₃₂	0.79	11.41	6.82	1.23	3.58
32 Cholesterol		0.82	-	0.59	0.47	0.23
33 Campesterol		0.89	1.69	2.52	2.23	1.81
34 Stigmasterol		0.92	5.74	6.35	6.66	6.02
35 B-sitosterol		1.00	8.32	14.43	12.51	13.18
Total hydrocarbon			84.25	76.11	78.13	78.76
Total Sterols			15.75	23.89	21.87	21.24

T : traces .

R.R.T. : Relative retention time

Table (4) : Effect of humid and dry heat treatments on chemical composition of rapeseeds meal .

Contents	Humid treatments				R e a s t i n g t r e a t m e n t s					
	Control	1 0 0 ° C			1 2 0 ° C		1 4 0 ° C			
		5 min.	10 min.	20 min.	30 min.	60 min.	30 min.	60 min.	30 min.	60 min.
Moisture	9.28	9.22	9.03	9.16	8.90	8.40	8.40	8.40	7.10	7.10
Ash	7.04	6.83	6.94	6.96	6.60	6.38	6.51	6.55	6.40	6.73
Crude fiber	8.46	7.57	7.46	7.39	7.52	7.37	7.13	7.53	7.36	7.24
Protein	34.63	34.28	34.11	34.17	34.15	33.89	33.46	33.72	33.81	33.86
Reducing and Non-reducing sugars	4.60	3.46	1.25	0.92	6.98	7.28	7.24	7.29	7.32	7.52
Minerals										
Na	0.022	0.022	0.022	0.021	0.021	0.019	0.019	0.020	0.021	0.020
K	0.44	0.41	0.40	0.42	0.44	0.44	0.42	0.43	0.40	0.41
Zn	0.16	0.150	0.146	0.154	0.160	0.150	0.150	0.140	0.160	0.160
Cu	0.037	0.036	0.035	0.036	0.038	0.036	0.036	0.037	0.036	0.035
Mn	0.080	0.079	0.075	0.077	0.079	0.077	0.078	0.081	0.078	0.079

Autoclaving treatments had no effect on protein amounts of the residual meal, while roasting caused a slight decrease in this material, table (4).

Element contents showed a minute changes upon the application of either autoclaving or roasting.

5- Effect of heat treatments on total glucosinolate compounds and myrosinase enzyme activity:

Table (5) showed that both total glucosinolate compounds and myrosinase enzyme activity had suffered slight decrease as a result of autoclaving treatments. On the other hand, roasting treatments induced a gradual decrease in both total glucosinolate compounds and myrosinase enzyme activity with increasing the temperature of roasting. Similar results were reported by Josefsson and Radwan and Lu (1976). It is important to mention that roasting at 140°C for 60 min. induced the highest reduction in total glucosinolate compounds i.e. 84.04% and had led to the highest inactivation effect on myrosinase enzyme activity i.e. the decrease in activity reached 94.32% when compared with other treatments. Such data might suggest that roasting had better effect on the inactivation of both total glucosinolate compounds and myrosinase enzyme than that of humid heat (autoclaving).

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تأثير الحرارة الرطبة والجافة على نوعية الزيت والكسب

المتخلف من بذور اللفت " الشلجم "

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خليل حلمى الخواص

تمت معاملة بذور اللفت " الشلجم " بالحرارة الرطبة تحت ضغط " الاوتوكلاف " وكذلك عوملت بالحرارة الجافة " التحميص " بهدف دراسة تأثير هاتين المعاملتين على كل من الزيت والكسب المتبقى فى محاولة لتحسين نوعيتها بالاضافة الى تشييت أنزيم الميروزينيز لتقليل المركبات السامة التى تنتج من تحليل بعض مركبات الجلوكوسنولتيه بفعل هذا الأثرزيم .

أدت الحرارة الحافة الرطبة الى تغيرات واضحة فى بعض الخواص الطبيعية والكيمائية فى الزيت والكسب . وقد تسببت هاتين المعاملتين فى بعض التغيرات فى النسب الكلية وكذلك مفردات - الأحمض الدهنية المشبعة والغير مشبعة .

وقد أوضحت النتائج أن هناك انخفاض واضح فى نسبة حمض الاربوسيك (C_{22}) الغير مرغوب قدره ٠.٦٨٧٧٪ نتيجة معاملة البذور بالحرارة الرطبة (الاتوكلاف) لمدة ٢٠ دقيقة .

تفريد الهيدروكربونات والاستيرولات للبذور المعاملة بهاتين المعاملتين بواسطة الكروماتوجرافى الغازي أوضح أن المركب (C_{24}) هو المكون السائد فى الهيدروكربونات . بينما المركب بيتاستيوستيرول B-sitosterol هو المركب الاساسي فى الستيرولات وقد ازدادت نسبته بدرجة ملحوظة نتيجة لهذه المعاملات .

أوضحت النتائج أن تأثير المعاملة الرطبة (الاتوكلاف) كان قليلا سواء على الجلوكوسينولات الكلية أو نشاط أنزيم الميروزينيز بالبذرة وعلى العكس من ذلك وقد أدت الحرارة الجافة (التحميص) الى انخفاضاً كبيراً فى كمية الجلوكوسينولات الكلية (٠.٨٤٠٤٪) فى حالة تحميص البذور على درجة ١٤٠م لمدة ساعة وكذلك تشييط نشاط أنزيم الميروزينيم الى حد كبير بتأثير ذات المعاملة (٠.٩٤٣٢٪) .