Deodorization of Vegetable Oils: Prediction of *trans* Polyunsaturated Fatty Acid Content

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ABSTRACT: Kinetics of the formation of *trans* linoleic acid and *trans* linolenic acid were compared. Pilot plant-scale tests on canola oils were carried out to validate the laboratory-scale kinetic model of geometrical isomerization of polyunsaturated fatty acids described in our earlier publication. The reliability of the model was confirmed by statistical calculations. Formation of the individual *trans* linoleic and linolenic acids was studied, as well as the effect of the degree of isomerization on the distribution of the *trans* fatty acid isomers. Oil samples were deodorized at temperatures from 204 to 230°C from 2 to 86 h. Results showed an increase in the relative percentage of isomerized linolenic and linoleic acid with an increase in either the deodorization time or the temperature. The percentage of *trans* linoleic acid (compared to the total) after deodorization ranged from <1 to nearly 6%, whereas the percentage of *trans* linolenic acid ranged from <1 to >65%. Applying this model, the researchers determined the conditions required to produce a specially isomerized oil for a nutritional study. The practical applications of these trials are as follows: (i) the *trans* fatty acid level of refined oils can be predicted for given deodorization conditions, (ii) the conditions to meet increasingly strict consumer demands concerning the *trans* isomer content can be calculated, and (iii) the deodorizer design can be characterized by the deviation from the theoretical *trans* fatty acid content of the deodorized oil.

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KEY WORDS: Deodorization, kinetics, linoleic acid, linolenic acid, modeling, *trans* isomer fatty acids.

Formation of geometrical isomers of polyunsaturated fatty acids during deodorization of vegetable oils is a well-known fact. In accordance with nutritionists' proposals, there is a strong tendency in Europe to keep the *trans* fatty acid isomer content of edible oils as low as possible. Concerning the isomerization of polyunsaturated fatty acids, Wolff (1) reported that the probability of linolenic acid isomerization is 12 to 15 times higher than that of linoleic acid. It has also been shown that geometrical isomerization of the linoleic and linolenic acids follows first-order kinetics (2,3). In spite of this finding, no kinetic studies comparing the isomerization of linoleic acid and linolenic acid have been conducted.

In the framework of the European Union project entitled "Nutritional and Health Impact of *trans* Polyunsaturated Fatty Acids in European Populations," the researchers investigated the metabolic effect of *trans* linolenic acid isomers. Fully refined oils containing extremely high amounts of *trans* linolenic acid (5%) and the lowest possible quantities of *trans* linoleic acid (max. 0.5%) were requested for the study. All other quality parameters had to meet fully refined oil specifications, as the isomerized oil was to be used in a human diet. In the oil requested, both the 5% *trans* linolenic acid isomer content and the ratio of the *trans* linolenic to the *trans* linoleic acid isomer contents are quite different from the values characteristic of commercially available refined oils. Therefore, a model for geometrical isomerization was established based on laboratory deodorization trials and presented in our earlier publication (4). The kinetics of *trans* linoleic acid and *trans* linolenic acid formation were described, taking into account the linolenic acid loss found during long-term heating.

For the pilot plant-scale study, the scientific objectives were as follows: (i) to confirm the reliability of the laboratory-scale model, (ii) to obtain more information on the rate of formation of the individual geometrical isomers, and (iii) to determine the exact conditions needed for the pilot-plant production of the above-mentioned specially isomerized oil.

Particular attention was paid to the practical use of the model, aiming at the following: (i) prediction of *trans* fatty acid isomer formation during industrial deodorizations, (ii) calculation of deodorization conditions to meet the expectations for the *trans* fatty acid content of edible oils, and (iii) characterization of the deodorizer design by assessing the deviation from the theoretical *trans* fatty acid content.

MATERIALS AND METHODS

Materials. Bleached canola oils for the pilot plant experiments were supplied by the Cereol refinery located at Martfű (Hungary) and Raision Margariini (Raisio, Finland). The fatty acid composition of the bleached oils is given in Table 1. Samples for the interlaboratory test were prepared at Cereol Group Center of Expertise, Cappelle la Grande, France.

Pilot plant-scale deodorization. The trials were conducted in the pilot-plant refinery of Cereol Group Center of Expertise (Budapest, Hungary). In this paper the term "deodorization" will be used for isomerization of oils under the specific conditions detailed below.

To check the reliability of the model, experiments were carried out in a 100-L batch deodorizer. The oil was heated to

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TABLE 1
Fatty Acid Composition (%) of Bleached Canola Oils

	A[a]	B[b]
16:0	4.8	3.1
16:1	0.2	0.1
18:0	1.8	1.7
18:1	60.1	58.5
18:2	21.3	21.9
18:3	8.3	11.9
20:0	0.6	0.5
20:1	1.3	1.0
22:0	0.4	0.3
22:1	0.4	0.1
24:0	—	0.2
24:1	—	0.3
Others[c]	0.8	0.4

[a]Bleached canola oil from Cereol Martfű (Martfű, Hungary).
[b]Bleached canola oil from Raision Margariini (Raisio, Finland).
[c]Unidentified peaks.

TABLE 2
Results of Interlaboratory Test on Determination of *trans* Fatty Acid Isomer Content (%)

	Sample 1[a]		Sample 2[b]	
	Lab 1[c]	Lab 2[d]	Lab 1[c]	Lab 2[d]
cis 18:2	21.43	21.18	20.54	20.60
trans 18:2	0.00	0.00	0.80	0.89
Total 18:2	21.43	21.18	21.34	21.49
cis 18:3	10.10	9.72	4.10	4.12
trans 18:3	0.00	0.00	5.30	5.38
Total 18:3	10.10	9.72	9.40	9.50

[a]Bleached canola oil.
[b]Isomerized canola oil.
[c]Measured by Cereol Group Center of Expertise, Cappelle la Grande, France.
[d]Measured by Cereol Group Center of Expertise, Budapest, Hungary.

the operating temperature under reduced pressure (<2 mbar), and nitrogen was applied as the stripping gas. A hot silicon liquid, Syltherm 800 (Dow Corning Corp., Midland, MI) was circulated in the equipment jacket for heating. Deodorization time was counted after the desired temperature had been reached. The accuracy of the temperature regulation was ±1°C. The continuous operations lasted 24, 48, and 86 h (oil A) and 67.5 h (oil B) at temperatures of 230, 220, 210, and 204°C, respectively. Samples were regularly taken at room temperature through a cooling sampling system.

Analysis of fatty acid composition. Fatty acid methyl esters (FAME) were prepared according to American Oil Chemists' Society Official Method AOCS Ce 2-66 (5) and were analyzed on a 5890 Series II Plus gas chromatograph (Hewlett-Packard) equipped with an electronic pressure control system, a split/splitless injector (210°C), and a flame-ionization detector set at 210°C. A fused-silica column 60 m × 0.25 mm i.d., 0.2 μm film coated with SP2340 (Supelco, Inc., Bellefonte, PA) was used for FAME analysis. The carrier gas was hydrogen at a constant flow rate of 20 cm/s. The oven temperature was programmed from 150 to 200°C at 1.3°C/min, then held at 200°C for 10 min. Quantitative analysis was performed with HP Chemstation 3365 software. Before starting the pilot-scale trials, this method was successfully cross-checked against the one applied for the laboratory experiments at the Cereol Group Center of Expertise, Cappelle la Grande (Table 2).

Tocopherol. Tocopherol content of the oils was determined according to the ISO 9936:1997 method, using high-performance liquid chromatography (6).

RESULTS AND DISCUSSION

The total linoleic acid content was constant in all the pilot deodorization trials, but a characteristic decrease in the amount of linolenic acid was found, as previously reported (6). During deodorization of oil A (lower initial linolenic acid content), 4.62, 5.04, and 5.02% of total *trans* linolenic acid isomers were achieved by the end of the process at 210°C/86 h, 220°C/48 h, and 230°C/24 h, corresponding to isomerization degrees of 60.8, 64.5 and 65.0%, respectively (Fig. 1). The isomerization degree (symbolized as DI_L or DI_{Ln}), expressed as a percentage, is the ratio between the total *trans* linoleic or linolenic acid isomer content and the corresponding total linoleic or linolenic acid content. The total *trans* isomer content for linoleic acid was 0.95, 1.01, and 1.23% at 210, 220, and 230°C, respectively, representing DI_L of 4.5, 4.7, and 5.8%.

In the case of oil B, which was richer in linolenic acid, 4.71% of the total *trans* linolenic acid isomers were detected after deodorization at 204°C for 67.5 h, which corresponded to a DI_{Ln} of 40.7%. Only 0.49% of the total *trans* linoleic acid isomers were detected, which represents a DI_L of 2.2%.

Statistical calculations. The reliability of the laboratory model for *trans* linoleic and *trans* linolenic acid formation was checked by statistical analysis. The differences between the values of *trans* fatty acid content measured in the pilot plant tests and those calculated using the theoretical model were analyzed. Student's *t*-test was applied to determine the significance of these differences. The Abbe test was also conducted to investigate whether there was a systematic order in the differences. Calculations were performed on samples heated at each temperature for both linoleic and linolenic acid.

Under pilot-plant conditions, the *trans* fatty acid content was found to increase slightly during the heating period. Also, the higher the temperature to be reached, the higher the amount of *trans* fatty acids formed. (No *trans* isomers were detected during heating to 210°C, but 0.1% *trans* linoleic and 0.5% *trans* linolenic acid isomers were found when heating to 230°C.) To eliminate this effect, only *trans* isomers formed after reaching the operating temperature were taken into consideration. To characterize the development of *trans* fatty acids formed strictly at the operating temperature, we transformed the measured *trans* fatty acid content as follows:

$$(\textit{trans}\ \text{C18:3})_t = (\textit{trans}\ \text{C18:3})_t - (\textit{trans}\ \text{C18:3})_{t=0} \qquad [1]$$

(and similarly for linoleic acid), where $(\textit{trans}\ \text{C18:3})_t$ is the measured *trans* linolenic acid content at time *t,* and $(\textit{trans}\ \text{C18:3})_{t=0}$ is the amount of *trans* linolenic acid at time $t = 0$.

The consequence of isomerization during the warmup

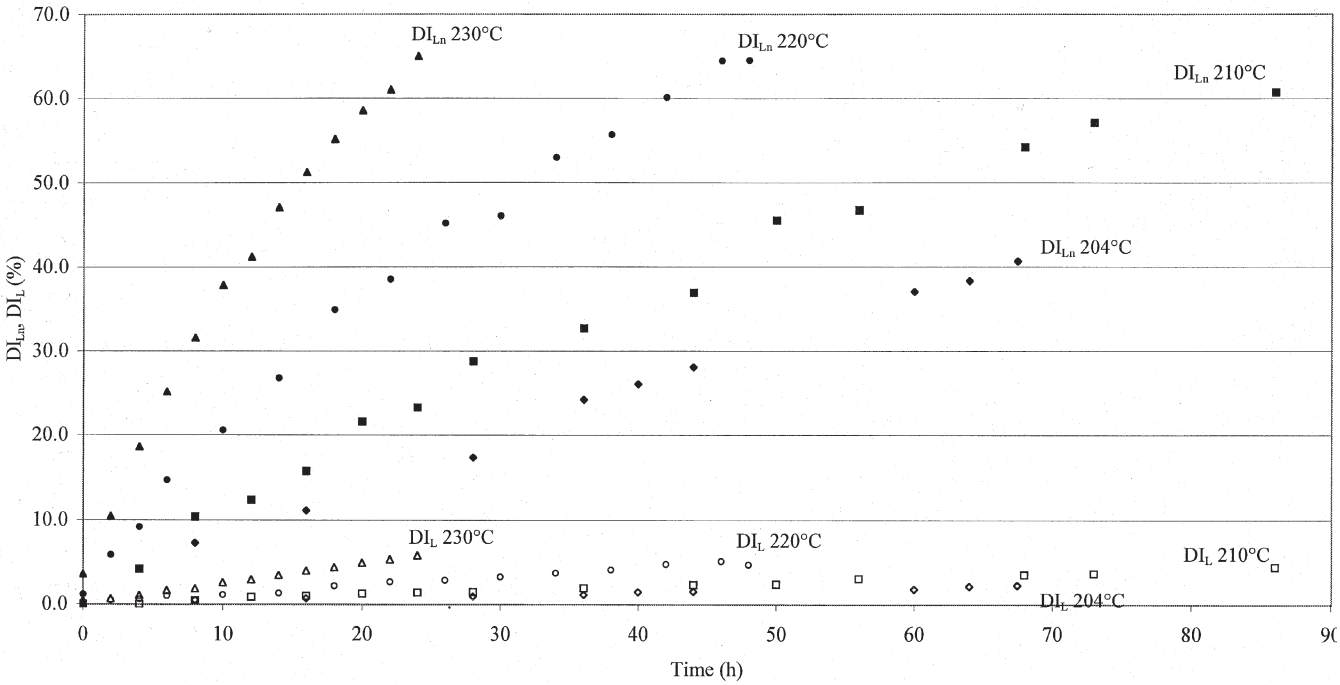

FIG. 1. Degree of isomerization of linolenic acid, DI_{Ln}, and linoleic acid, DI_L, as a function of time (pilot-scale deodorization of canola oil). Abbreviations: DI_{Ln} is defined as the percentage of the total *trans* linolenic acid content over the total linolenic acid content; DI_L is defined as the percentage of the total *trans* linoleic acid content over the total linoleic acid content.

phase was that by the time the operating temperature ($t = 0$) was reached, the concentrations of *cis* linoleic and linolenic acids had begun to decrease. This decrease can be compensated for by using the following fatty acid composition for theoretical calculations:

$$(cis\ \text{C18:3})_{t=0,\text{theo}} = (cis\ \text{C18:3})_i - (trans\ \text{C18:3})_{t=0} \quad [2]$$

and

$$(trans\ \text{C18:3})_{t=0,\text{theo}} = 0 \quad [3]$$

thus

$$(\text{total C18:3})_{t=0,\text{theo}} = (cis\ \text{C18:3})_i - (trans\ \text{C18:3})_{t=0} \quad [4]$$

(and similarly for linoleic acid), where $(cis\ \text{C18:3})_i$ is the measured initial *cis* linolenic acid content before deodorization, $(cis\ \text{C18:3})_{t=0}$ is the measured *cis* linolenic acid content at time $t = 0$, $(cis\ \text{C18:3})_{t=0,\text{theo}}$ is the theoretical *cis* linolenic acid content at time $t = 0$, and $(\text{total C18:3})_{t=0,\text{theo}}$ is the theoretical total linolenic acid content at time $t = 0$.

By performing Student's *t*-test with the above considerations, the value of $t_{0.05}$ was lower than the critical *t* value in each case. In other words, the mean of the difference between the measured and the theoretical values was zero with 95% confidence at all the examined temperatures for both linoleic and linolenic acid. For example, at 230°C the mean of the differences between the theoretical and measured *trans* linolenic acid content was –0.01% with a standard deviation of 0.04% (number of measurements, $n = 13$). The above data resulted in $t_{0.05} = 0.66$, which is lower than the critical *t* value of 2.16. The Abbe test proved the absence of a systematic order in the differences at a confidence level of 95% in all cases, as the values of the test statistic $R_{0.05}$ were greater than the critical *R* values. In the test at 230°C, $R_{0.05}$ was 0.72 for linolenic acid, compared to the critical *R* value of 0.61. Therefore, the model provided accurate information on *trans* fatty acid isomer formation.

When *trans* isomers are already present before reaching the operating temperature, the DI of deodorized oil can be predicted as follows:

$$\text{DI}_{\text{Ln}} = \frac{(trans\ \text{C18:3})_{t,\text{theo}} + (trans\ \text{C18:3})_{t=0}}{(\text{total C18:3})_{t,\text{theo}} + (trans\ \text{C18:3})_{t=0}} \quad [5]$$

The equation simplifies when no *trans* isomers are detected at $t = 0$, which is generally the case in industrial circumstances.

Distribution of the individual geometrical isomers. Four *trans* isomers of linolenic acid were detected in the samples taken during the pilot-plant deodorization tests. (The abbreviations *c* and *t* are used here for *cis* and *trans* configurations, respectively.) The two main isomers were *c,c,t* and *t,c,c* linolenic acids, and the two minor isomers were *c,t,c* and *t,c,t* linolenic acids, as reported previously (4,8,9).

Some surveys have shown that the distribution pattern of these geometrical isomers varies within a very narrow range in commercial edible oils (2,8) and only limited data are available on its dependence on the degree of isomerization (9).

In Figure 2, the relative amounts of individual geometrical isomers of linolenic acid are plotted as a function of DI. A linear relationship was found for each individual isomer in a range limited to DI lower than 35%. In the case of *c,c,t; t,c,c*; and *t,c,t* isomers, a strong correlation was observed.

The relative amounts of the two main components—*c,c,t* and *t,c,c*—decreased slightly, whereas that of the di-*trans* isomer *t,c,t* increased with increasing DI_{Ln}. The relative

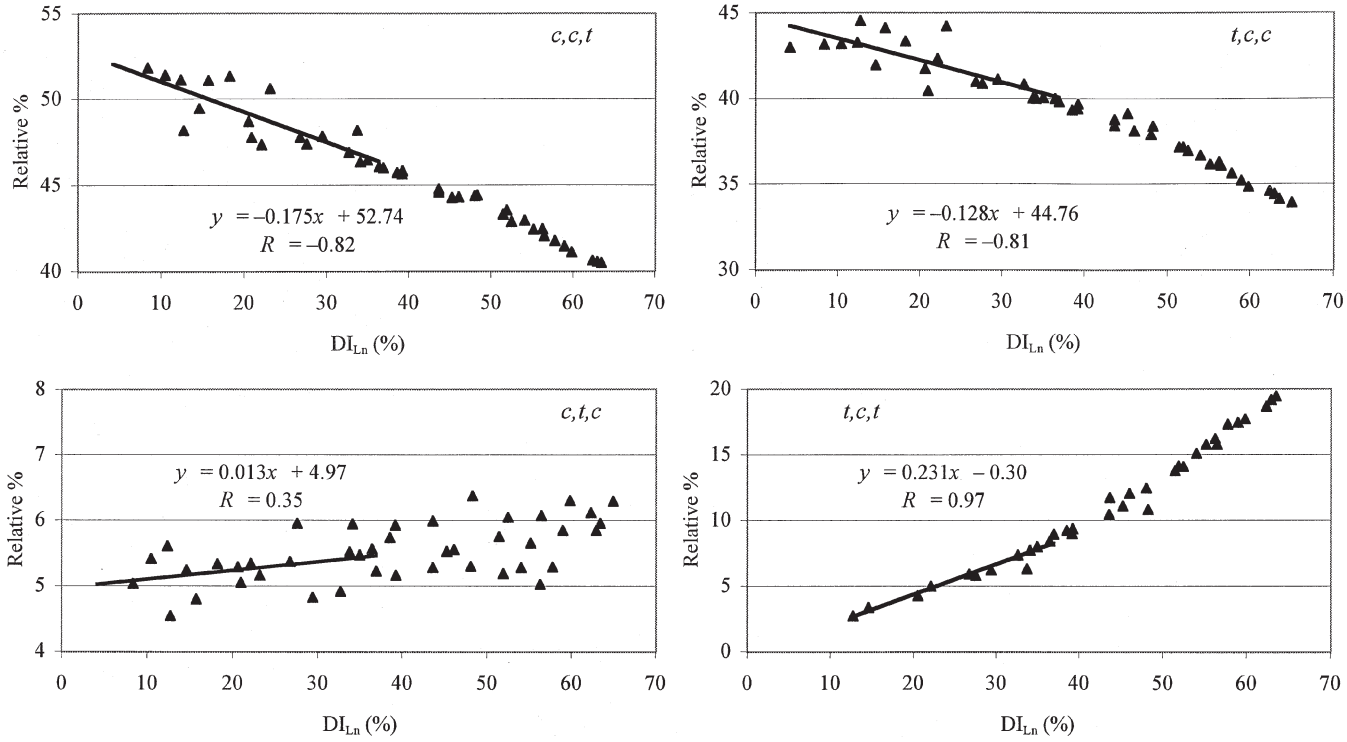

FIG. 2. Distribution of *trans* C18:3 isomers as a function of degree of isomerization, DI_{Ln} (pilot-scale deodorization of canola oil). For abbreviation, see Figure 1.

percentage of the *c,t,c* isomer remained nearly constant to a DI_{Ln} of 65% (the end point of our experiments). Extrapolation to DI = 0 indicated that at the beginning of the reaction, the probability of formation of individual geometrical isomers was 51.5, 43.6, 4.9, and 0% for the *c,c,t; t,c,c; c,t,c;* and *t,c,t* isomers, respectively, which corresponds closely with the pattern described by Wolff (8).

Because DI_{Ln} increased with the temperature and the duration of the deodorization, it is useful to consider the effect of operating conditions on the relative amounts of the abovementioned isomers (Fig. 3). For example, under mild conditions (210°C, 4 h) only the two main isomers—*c,c,t* and *t,c,c*—were detected. When more general industrial deodorization conditions (230°C, 2 h) were applied, the four isomers appeared in a distribution pattern comparable to that of commercially available refined oils (1,8). Under extreme circumstances

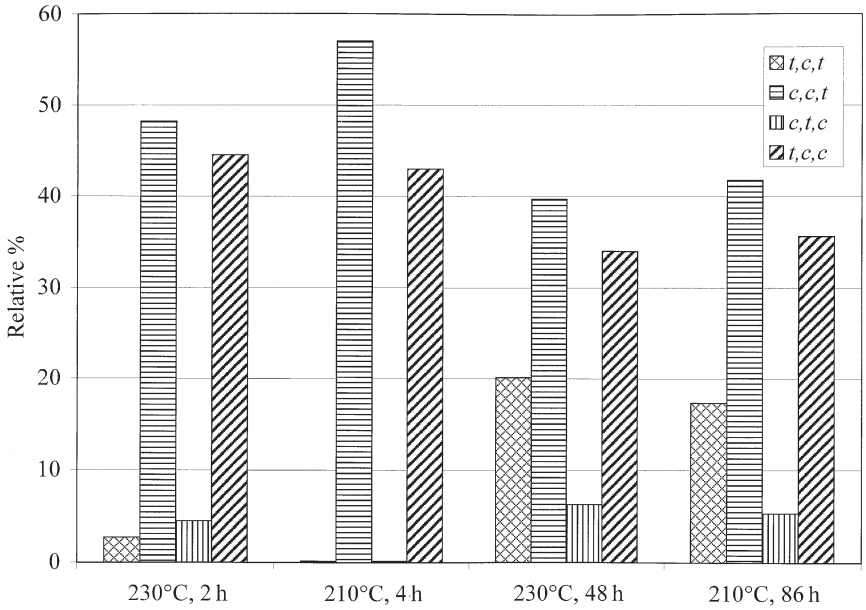

FIG. 3. The effect of deodorization conditions on distribution of individual *trans* C18:3 isomers (pilot-scale deodorization of canola oil).

(230°C, 48 h or 210°C, 86 h), the relative percentage of the *t,c,t* isomer increased, resulting in a decrease of *c,c,t* and *t,c,c* components. The *c,t,c* isomer showed no noticeable change.

Concerning the isomerization of linoleic acid, two mono-*trans* isomers—*c,t* and *t,c*—were detected. At DI_L up to 1.2%, the relative amounts of the *c,t* isomer were greater than those reported for commercial edible oils (8,9). The initial probability of its formation was twice that of the *t,c* isomer. With increasing DI, the amounts of these two compounds became closer to each other. At DI ≥4% the relative amounts of each *c,t* and *t,c* isomer were 51 to 52 and 49 to 48%, respectively (Fig. 4).

The relative probability of isomerization of linolenic and linoleic acids during industrial deodorization was expressed by Wolff (1) as the ratio between the degree of isomerization of the two fatty acids (DI_{Ln}/DI_L). This ratio depends on temperature and time of deodorization, and it is relatively stable (ranging from 12 to 15) for edible oils (2,8). The relative probabilities calculated from the kinetic model were in a similar range. For example, the calculated values for 2 h at 230°C, 1 h at 245°C, and 0.5 h at 255°C were 16.3, 14.8, and 13.6, respectively.

Production of selectively isomerized oil for a nutritional study. The conditions necessary to produce selectively isomerized oil were then calculated using the model. The term "selective isomerization" is used because an uncommon ratio of the *trans* linolenic acid content over the *trans* linoleic acid content (*r*) was aimed at. The objective was to obtain a *trans* linolenic acid content as high as 5% and to achieve a ratio of $r = 10$.

For pilot plant-scale deodorization, a bleached canola oil containing 11.9% linolenic acid and 21.9% linoleic acid and deodorization conditions of 204°C and 74 h were chosen as an acceptable compromise between the expected *trans* fatty acid composition and the deodorization time realizable in pilot-plant conditions. As the temperature is increased, the time of the operation is much shorter (13.5 h at 230°C for the same oil), but the ratio between the *trans* linolenic and *trans* linoleic acids (*r*) drops below an acceptable level ($r_{204°C} = 9.6$, $r_{230°C} = 7.2$). The initial fatty acid composition of the raw material also has a basic influence on the necessary deodorization time. The higher the initial linolenic acid content is, the shorter the operation. Because the bleached oil contained 10% initial linolenic acid, 96 h was necessary to reach 5% *trans* linolenic acid at 204°C with an *r* value of 7.4. An improvement in this ratio by reducing the temperature would require an excessively long deodorization time, which is not feasible even on a pilot-plant scale. Three batches of selectively isomerized oil were produced, each with a *trans* fatty acid profile very close to that predicted by the theoretical model (Table 3). The *trans* linoleic and *trans* linolenic acid contents were 0.56 and 5.12% on average, which is close to the target values of 0.5 and 5.0%. To provide sufficient reference material for the nutritional study, "zero *trans*" canola oil was produced on a pilot-plant scale. No *trans* fatty acid isomers were detected after deodorization at 175°C for 4.5 h (Table 3). The observations available in the literature indicate the following: (i) a slow rate of isomerization at 180 to 200°C (10), (ii) no *trans* isomers detected after deodorization at 180°C for 20 h (4), and (iii) 3% of the linolenic acid isomerized during 16 h at 190°C (9). Concerning organoleptic properties, both the isomerized and the reference oils had a neutral taste and odor. No remarkable change was noticed in the tocopherol content after deodorization at 175°C for 4.5 h (710 mg/kg was measured in the bleached oil and 700 mg/kg in the deodorized oil). A still-acceptable 15% decrease was detected during the long-term treatments at 204 to 205°C, where the total tocopherol content dropped to 600 mg/kg on average (Table 3).

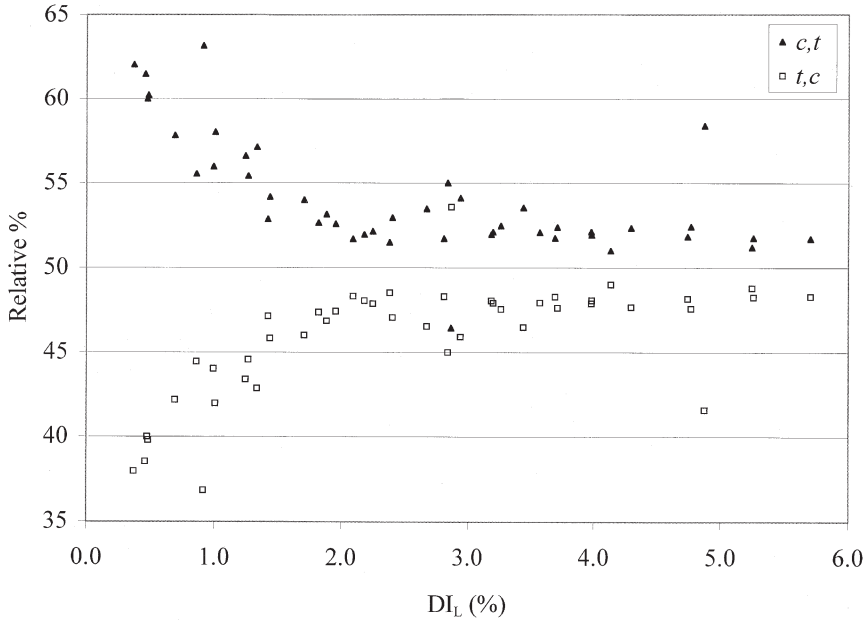

FIG. 4. Relative amount of individual *trans* C18:2 isomers as a function of degree of isomerization, DI_L (pilot-scale deodorization of canola oil). For abbreviation, see Figure 1.

TABLE 3
Quality of Pilot Scale-Produced Selectively Isomerized and "Zero *trans*" Canola Oils: Comparison of Experimental and Theoretical *trans* Fatty Acid Contents[a]

	Temp. (°C)	Time (h)	*trans* C18:2 (%) Meas.[b]	*trans* C18:2 (%) Theo.[c]	*trans* C18:3 (%) Meas.[b]	*trans* C18:3 (%) Theo.[c]	Peroxide value (meq/kg)	T_{420}[d] (%)	Tocopherols (mg/kg) α	β	γ	δ
Bleached oil			ND[e]	ND	ND	ND	4.7	3.6	230	ND	460	20
Selectively isomerized oil, batch 1	204	74	0.55	0.52	5.07	5.02	0.1	73.4	159	ND	397	18
Selectively isomerized oil, batch 2	204	67.5	0.49	0.48	4.71	4.70	0.3	71.3	174	ND	409	19
Selectively isomerized oil, batch 3	205	82	0.63	0.63	5.58	5.63	0.2	74.6	161	ND	380	17
"Zero *trans*" oil	175	4.5	ND	0.00	ND	0.05	0.2	30.6	230	ND	450	20

[a]Initial total linoleic acid content: 21.9%; initial total linolenic acid content: 11.9%.
[b]Measured from the pilot scale-produced oil.
[c]Theoretical value calculated from the model.
[d]Transmittance at 420 nm.
[e]ND, not detected.

Prediction and control of the trans *polyunsaturated fatty acid level in industrial deodorization.* It is of importance to the oil industry to have a model available to predict the *trans* polyunsaturated fatty acid content of deodorized oils. As an example, to keep the DI_{Ln} under 10% and the DI_L under 0.7%, the deodorization time and especially the temperature must be limited; approximately 240°C for 1 h or 230°C for 2.5 h can be calculated. According to one survey of European edible oils, DI_{Ln} varied in a wide range from 10.5 to 26.9% (9). Approximately 40% of the survey samples had a DI_{Ln} between 15 and 20%, and only 20% had a DI_{Ln} lower than 10%.

In order to check the formation of *trans* fatty acid isomers during the industrial process, sunflowerseed, rapeseed, and soybean oil samples were collected from six deodorizers, A to F (both before and after deodorization). The samples were taken during stable operation of the deodorizers under well-known conditions (temperature, duration, pressure, and stripping steam dosage), and their *trans* fatty acid contents were analyzed. The DI of linoleic and linolenic acids were calculated in such a way that only the *trans* isomers formed during deodorization were taken into consideration. These numbers were compared with the theoretical values (Table 4). A good correspondence was found in the case of five of the six deodorizers. Only for deodorizer D was the measured *trans* isomer level systematically much higher than predicted, suggesting that the deodorizer design had an effect. The possible causes could include local overheating, inhomogeneous heating, or liquid holdup (heterogeneous residence time) that may

TABLE 4
Isomerization Level of Industrially Deodorized Refined Oil Samples, Comparison with the Theoretical Values

Oil type	Deodorizer	Temp. (°C)	Time (h)	DI_L[a] (%) Ind.[c]	DI_L[a] (%) Theo.[d]	DI_{Ln}[b] (%) Ind.	DI_{Ln}[b] (%) Theo.
Sunflower	A	200	1.5	0.06	0.04	—	—
Sunflower	A	210	2.0	0.14	0.11	—	—
Sunflower	A	227	1.5	0.24	0.28	—	—
Sunflower	B	223	2.0	0.24	0.28	—	—
Sunflower	B	223	2.0	0.23	0.28	—	—
Sunflower	B	225	3.5	0.73	0.57	—	—
Sunflower	C	223	2.0	0.24	0.28	—	—
Sunflower	D	222	2.0	1.01	0.26	—	—
Sunflower	D	225	2.0	1.15	0.33	—	—
Sunflower	E	238	1.5	0.76	0.62	—	—
Sunflower	E	240	1.5	0.91	0.72	—	—
Canola	B	215	2.0	0.40	0.15	1.93	3.23
Canola	B	220	3.5	0.37	0.39	6.04	7.64
Canola	C	200	2.0	0.00	0.05	0.73	1.18
Canola	C	226	2.0	0.21	0.35	5.10	6.43
Canola	F	243	2.0	1.30	1.17	18.52	17.04
Canola	F	246	2.0	1.69	1.43	20.82	19.96
Soy	C	210	2.0	0.09	0.11	1.57	2.33
Soy	C	226	2.0	0.37	0.35	5.50	6.43
Soy	C	227	2.0	0.33	0.38	5.70	6.83
Soy	D	225	2.0	1.50	0.33	20.70	6.05

[a]DI_L is defined as the percentage of the total *trans* linoleic acid content over the total linoleic acid content.
[b]DI_{Ln} is defined as the percentage of the total *trans* linolenic acid content over the total linolenic acid content.
[c]Measured from the industrial sample.
[d]Theoretical values from the model.

have resulted in a positive deviation from the theoretical values (a higher DI than the theoretical). Devinat *et al.* (11) also reported a significant effect from the deodorizer geometry on geometrical isomerization.

Apart from equipment design, several other sources of deviation have to be taken into consideration. For example, internal recirculation of the oil may cause a higher *trans* isomer level than expected. Also, at an industrial level, a reliable and representative temperature measurement in the bulk of the oil is more difficult to obtain than in a pilot installation.

The above data demonstrate that the model could be used to characterize *trans* linoleic and *trans* linolenic acid formation in the industrial process. Accurately characterizing this process requires a thorough sampling and a precise knowledge of the process conditions; in the cases where a considerable positive difference is found, it is advisable to check the deodorizer.

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