TAGUCHI'S METHODOLOGY FOR DETERMINING OPTIMUM OPERATING CONDITIONS IN HYDROTHERMAL PRETREATMENTS APPLIED TO CANOLA SEEDS

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The aim of the present work was to determine the optimum operating conditions in hydrothermal pretreatments applied to canola seeds. The effect of these pretreatments on the canola oil extraction yield and quality was evaluated. Samples were characterized by proximate analysis. Acidity and peroxide value were studied as parameters for the determination of oil quality. Seeds were exposed to direct steam contact in an autoclave. Pretreatments were carried out using different temperatures (100, 120 and 130 °C), exposition times (5, 15 and 30 min) and seed granulometry (ground seeds, with a particle size in a range from 0.420 to 1.000 mm; broken seeds, with a particle size ranging from 1.000 to 1.410 mm, and entire seeds). After each hydrothermal pretreatment, oil extraction was carried out by the Soxhlet method (hexane). The Taguchi method was followed in order to explore the optimum operating conditions by using an L9 experimental design, and select the most favourable levels of each variable. Initial oil content was 44.2% dry basis (db). The selected optimum experiment, using a temperature of 120 °C, a time of 5 min and broken seeds, generated an oil yield increase of 20% compared with non-hydrothermally treated seeds, whereas quality parameters remained within the accepted values for trade standards.

Keywords: Taguchi's methodology, hydrothermal pretreatment, canola, oil yield and quality

INTRODUCTION

Canola oil has beneficial health properties as it is low in saturated fats and contains a significant amount of essential fatty acids. On the other hand, this oilseed can also be used as a raw material for the production of alternative fuels (biodiesel) due to its high oil content, high yield per hectare and the good quality of its oil.[1] Presently, solvent extraction is the most commonly used method to recover oil from oilseeds. This process is controlled by mass transfer phenomena involving taking the oil from inside the lipid bodies to the solvent. The oil is inside the membrane, so it is necessary to break the cellular structure to let the oil be more accessible to the solvent. Pretreatments are applied to seeds in order to modify or break this structure, thus facilitating the liberation of oil.

The effect of vaporization on the degradation of the cellular structure of canola seeds during the conditioning step was studied. Improved mechanical properties were obtained due to this pretreatment, and thus, at industrial level, the pressing stage prior to solvent oil extraction was facilitated.[2] Hydrothermal pretreatments have also been reported as a means to obtain an efficient dehulling of canola seeds.[3] Mohamadzadeh et al.[4] studied the effect of the hydrothermal pretreatment on dehulling efficiency, and the quality and yield of the extracted oil from canola seeds. They found that the high moisture levels in the seeds over time affected the oil quality, promoted the hydrolysis of triglycerides that in turn generated free fatty acids, and reduced the shelf life of the oil as a result. On the other hand, when they analysed the peroxide index of the hydrothermally treated samples and that of the untreated samples, the differences were not significant. Similarly, Singh et al.[5] cooked crambe seeds with steam at 90–120 °C for 5–20 min in order to improve oil yield by pressing. In other case, the effect of hydrothermal pretreatments on the antioxidant activity in the oil obtained by pressing was evaluated by Szydlowska-Czerniak et al.[1] The use of steam in pretreatments of soybean has been widely reported, incorporating expanders in oil-processing plants. In some cases, this equipment can also be used in canola and sunflower processing in order to improve the mechanical and extraction properties of the seeds.[6,7]

The Taguchi methodology was initially designed to improve the quality of manufactured goods, and has since been applied in different research fields. It has been proved to be efficient in optimising several processes such as the optimisation of variables in ultra-precision machines for optical purposes, preparation of catalyst materials and metal extraction processes.[8–10] More recently, it has been used in the synthesis of substances such as TCH, for improving the recovery of metals from wastes and the removal of lead from bearing-lead anode slime.[11–13] In the edible oil extraction field, this method has been applied in SC fluid extraction of seed oil.[14] However, to the best of our knowledge, it has not yet been applied to optimise vegetable oil extraction pretreatments. If interactions among design parameters are neglected and error analysis is performed using approximation techniques instead of running additional experiments, the Taguchi method requires a significantly small number of experiments compared with other statistical techniques.[8]

The purpose of the present work was to determine the optimum operating conditions in hydrothermal pretreatments using steam

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by means of the Taguchi method, in order to study its effect on oil extraction yield and quality.

MATERIALS AND METHODS

Sample Characterization

Tests were carried out using canola seeds (one 5 kg bag) of the Barrel variety (grown in Argentina). The untreated sample was characterized by proximate analysis: levels of moisture (ASAE S 352.2 DEC.),[15] protein (AOCS Ai 4-91)[16] and lipid content (IUPAC 1.122).[17] Oil was characterized by determining the acidity value (IUPAC 2.201),[17] the peroxide index (AOCS Cd 8-53),[16] the tocopherol content by HPLC (AOCS Ce 8-89),[16] using α-tocopherol as standard[18] and fatty acid composition by gas chromatography.[19]

Hydrothermal Pretreatments

Seeds were subjected to hydrothermal pretreatments using steam in an autoclave (VZ, Argentina) whose base was perforated in order to facilitate the generation of steam from the bottom of the devise. The samples were placed on trays with a metallic mesh base. Then they were dried at 25 °C in a forced-circulation tunnel dryer to a moisture level of 6.5–7.4% (db). The hydrothermal pretreatments were carried out at different temperatures (100, 120 and 130 °C), exposition times (5, 15 and 30 min) and granulometry of the seeds (ground seeds, with a particle size in a range from 0.42 to 1.00 mm; broken seeds, with a particle size ranging from 1.00 to 2.00 mm, and entire seeds). The selected conditions of temperature and time were chosen based on previous preliminary studies carried out by our research group, and with the aim of extending the studied range of those variables.[20] The conditions analysed by Singh et al. for cooking crambe seeds with steam prior to the pressing stage ($T = 90$–120 °C, t = 5–20 min,[5] which in the present work were extended to 130 °C and 30 min) were also taken into account.

Experimental Design

The Taguchi methodology was used in order to explore the pretreatment operating conditions, and to select the optimum levels of each variable, using the L9 experimental design shown in Table 1.[21] A geometric representation of the design is shown in Figure 1. This design requires nine experiments, and although it does not have enough degrees of freedom to study interaction in systems with 3 factors at three levels, it was selected because it requires a low number of experiments. Interaction could be analysed only if an orthogonal array with a larger number of experiments was used, reducing the advantages of the Taguchi method. The experimental design was replicated.

Table 1. L9 orthogonal array

	Factor levels			
Experiment	A	B	C	D
E1	1	1	1	1
E2	1	2	2	2
E3	1	3	3	3
E4	2	1	2	3
E5	2	2	3	1
E6	2	3	1	2
E7	3	1	3	2
E8	3	2	1	3
E9	3	3	2	1

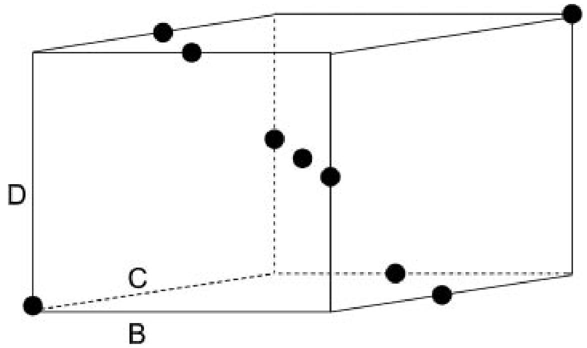

Figure 1. Experimental design.

It should be noted that Taguchi's definition of orthogonality is different from the usual chemometrical definition. Algebraic orthogonality could be reached by changing the factor levels 1, 2 and 3 in Table 1 to -1, 0 and 1, respectively.

Factors were assigned to columns B, C and D of Taguchi's matrix (Table 1). Column A was not considered in order to obtain an experimental design of Resolution IV;[22] that is to say, the main effects can be estimated, and they are not confused with each other or with two-factor interactions.

Table 2 shows the factors and levels used in the experimental design detailed in Table 1. It is worth noting that factor A is not included because it was not considered in the experiments. As mentioned above, only the last three columns of Table 1 were used.

In order to maintain the independence of the unknown and uncontrollable external factors that could affect the outcomes, the sequence of trials was executed at random according to the table of random numbers[21] in the following order: E3, E9, E8, E5, E6, E1, E7, E2 and E4.

Oil Yield and Quality Measurements

Each hydrothermally pretreated sample was ground, then the oil was extracted (IUPAC 1.122),[17] and quality measurements regarding the acidity value (IUPAC 2.201)[17] and peroxide index (AOCS Cd 8-53)[16] were carried out.

RESULTS AND DISCUSSION

Sample Characterization

Table 3 shows the characterization of the untreated sample in terms of moisture, lipid and protein content, expressed in dry basis (db). The moisture content of the seeds at harvest was in the range of 7.0–7.2% db. The oil content was 44.2% db, which was in the expected 40–60% db range for this species.[23] The mean protein content was 18.7% db, corresponding to a 33.5% db protein content in defatted meal, which is close to the range found in Argentinean canola meals (37–40%).[24]

Table 2. Experimental factors and levels

	Temperature (°C)	Time (min)	Granulometry
Level	B	C	D
1	100	5	Ground seeds
2	120	15	Broken seeds
3	130	30	Entire seeds

Table 3. Characterization of canola seeds

Determination (% db)	Canola
Moisture	7.1 ± 0.1
Oil	44.2 ± 0.8
Protein	18.7 ± 0.6

Table 4 shows the characterization of the oil extracted from untreated canola seeds in terms of acidity, peroxide index, tocopherol content and fatty acid composition. The polyene index (PI) is a measure of the susceptibility of the oil toward oxidation; PI is calculated as the ratio of polyunsaturated (PUFA) to saturated (SFA) fatty acids.

The fatty acid composition was in the expected range according to the literature for high oleic canola oil.[6,25]

Hydrothermal Pretreatments

Figure 2 shows the results of the ratio of oil yield from pretreated seeds to oil yield from untreated seeds (oil yield ratio). Figures 3 and 4 show data of the acidity (% oleic) and peroxide index (meq O_2/kg oil) quality parameters of the oil extracted from hydrothermally pretreated seeds, respectively.

The effect of the hydrothermal pretreatment was different depending on the operating conditions used. An increase of over 20% in oil yield was observed in treated samples (Figure 2) compared with the untreated sample. Mohamadzadeh et al. also observed an increase in oil yield due to the hydrothermal pretreatments applied to canola seeds (steamed and autoclaved) when they treated the seeds prior to dehulling.[4]

As it can be observed in Figure 3, the acidity value was affected by the hydrothermal pretreatments, being higher than that obtained for oil extracted from untreated canola seeds (0.54%). However, in all cases, these values were below 2%which is the maximum level permitted for crude oil by specifications of the Trading Rules of COPA (Canadian Oilseed Processors Association). A similar trend was observed by Mohamadzadeh et al. when they steamed or autoclaved entire seeds for 5–10 min as a pretreatment to dehulling, reaching acid values of 0.60–0.80 (% of oleic acid).[4] In contrast, Zacchi et al. used a steam treatment on rapeseeds at 100 and 145 °C, obtaining, in general, a better acidity index than in untreated samples.[26]

In regard to the peroxide index (Figure 4), an increase was observed in all the tests compared with the corresponding value of the oil extracted from the untreated sample, since peroxides were not detected in the untreated sample (Table 4). Nevertheless, oils from pretreated seeds showed a peroxide value below the maximum level established by Codex Alimentarius for refined oils (10 meq/kg) and for cold-pressed and virgin oils (15 meq/kg). In comparison, this value was not affected in steam-pretreated entire canola seeds at atmospheric pressure, nor in autoclaved ones at 100 kPa.[4] In this study, the pretreatments applied to entire seeds originated fewer changes in peroxide value (E3, E4 and E8) than in the rest of the essays, which could be due to the hull hindering the access of oxygen to the oil inside the seed. In addition, an effect of temperature could be observed among these three essays since the experiment carried out at the lower temperature produced the lowest peroxide content, and the one performed at the higher temperature generated the highest peroxide value. At higher temperatures than examined here, Zacchiet al.[27] found differences when canola seeds were autoclaved at 145 °C, reporting values up to 9.9 meq/kg, higher than those observed in this work, corroborating the influence of temperature.

Table 4. Characterization of the oil extracted from untreated canola seeds

Determination	Canola
Acidity (% oleic)	0.54 ± 0.09
Peroxide index (meq O_2/kg oil)	ND
Tocopherols (μg/g oil)	453
Fatty acid composition	
C16:0, palmitic acid (%)	4.23 ± 0.09
C18:0, stearic acid (%)	1.80 ± 0.06
C18:1, oleic acid (%)	72.21 ± 0.13
C18:2, linoleic acid (%)	15.17 ± 0.03
C18:3, linolenic acid (%)	6.55 ± 0.09
PUFA (%)	21.7 ± 0.12
SFA (%)	6.0 ± 0.15
PI = PUFA/SFA	3.6 ± 0.07

PI, polyene index; PUFA, polyunsaturated fatty acids; SFA, saturated fatty acids; ND, not detected.

Contact with oxygen in the air is essential for the oxidation process to proceed. This process is favoured when experiments are carried out using broken or ground seeds because a higher content of oil is exposed to oxygen. However, when entire seeds are used, the seed coat acts as a protective barrier against the degradation that the external medium could generate. In the present work, the oil quality parameters were less affected in the tests carried out with entire seeds (E3, E4 and E8 in Figures 3 and 4) than in the rest of the tests. Nevertheless, pretreatments applied to entire seeds did not produce a significant increase in oil yield (E3, E4 and E8 in Figure 2).

Sources of Variation

The Taguchi method uses the signal-noise ratio (SN) in order to explore the sources of variation. The term "signal" represents the desirable value (mean) for the output characteristic, and the term "noise" represents the undesiderable valuefor the output characteristic, that is to say the magnitude of the mean of a process compared to its variation. Since SN, expressed in decibels, is a measure of the variation within the experimental essays when noise factors are present, Taguchi used this amount to quantify the quality characteristics deviating from the desired value.[11]

For the problem of maximization of the oil yield ratio, the SN ratio was evaluated by means of Equation (1).[9,12]

$$SN = -10.\log\left[\frac{\sum_{k=1}^{n} 1/{x_k}^2}{n}\right] \tag{1}$$

In the case of the minimization problem (to obtain minimum values of acidity and peroxide index), the SN ratio was calculated using Equation (2).[9,12]

$$SN = -10.\log\left[\frac{\sum_{k=1}^{n} {x_k}^2}{n}\right] \tag{2}$$

where x_k is the response obtained experimentally (Figures 2–4), n is the number of measurements for each experiment, and k is the number of repetitions.

Table 5 shows values of the SN ratio calculated from responses of oil extraction yield and quality (acidity value and peroxide index).

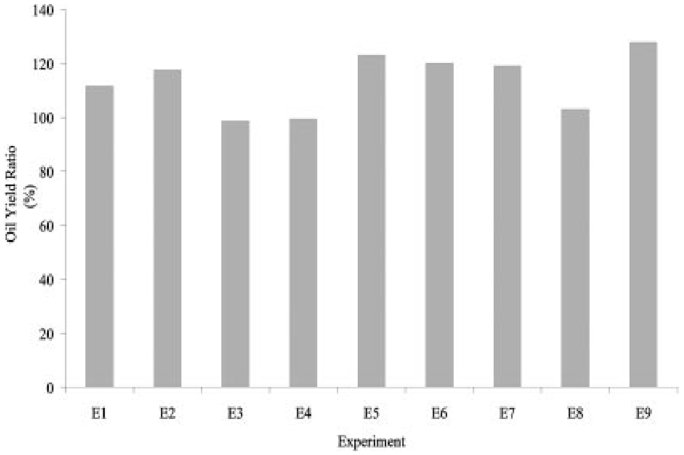

Figure 2. Ratio of oil yield of pretreated to untreated seeds (E1–E9 according to Table 1).

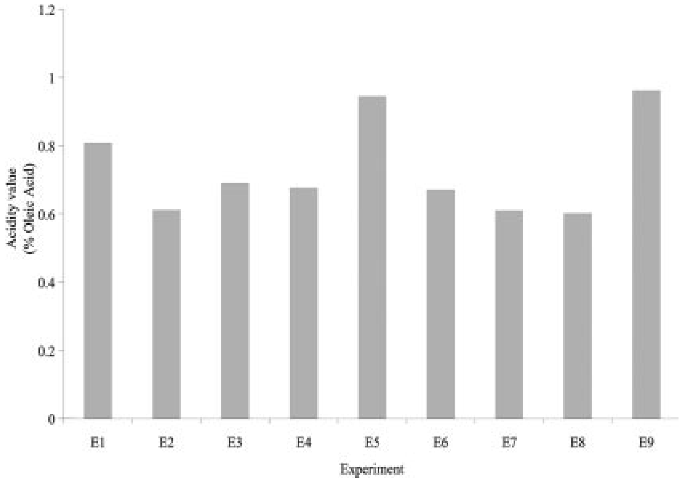

Figure 3. Acidity value of the oil obtained from hydrothermally pretreated canola seeds (E1–E9 according to Table 1).

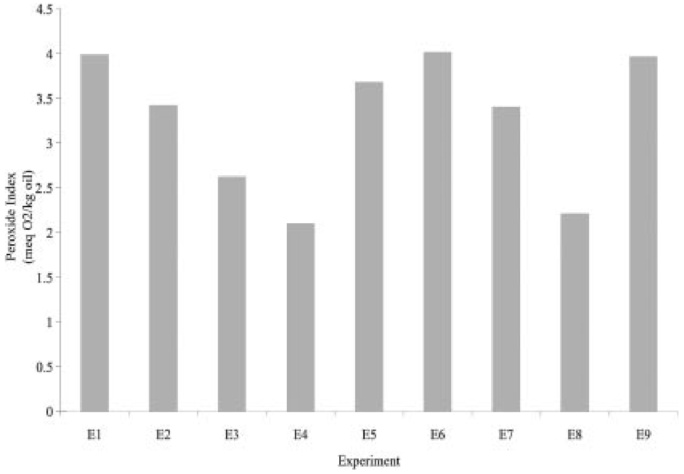

Figure 4. Peroxide index of oil obtained from hydrothermally pretreated canola seeds (E1–E9 according to Table 1).

In order to test the existance of significant interactions among the parameters, Taguchi suggests a "no interaction" model. Assuming no interactions, the effect of parameter i on SN can be found by comparing the average responses (M_{ij}) obtained at different levels j.[27] For example, the average response of D at level 1 in the oil yield analysis was obtained from the results of experiments 1, 5 and 9 (in Table 1, the level of D is equal to 1 in experiments 1, 5 and 9).

$$M_{\mathrm{D1}} = \frac{41.0 + 41.9 + 42.1}{3} = 41.7 \tag{3}$$

Values 41.0, 41.9 and 42.1 correspond to the signal-noise ratio of factor D (Table 5) for experiments E1, E5 and E9, in which the level of factor D is equal to 1 in the oil yield ratio analysis. Average responses (M_{ij}) of the parameters at the three levels are shownin Tables 6–8 for oil yield ratio, acidity value and peroxide index analysis, respectively.

The data shown in Tables 6–8 of the effects of the three control factors with their corresponding levels are graphically shown in Figures 5–7. Since a higher SN ratio indicates a better response (for example, higher oil yield ratio or lower acidity value and peroxide index), the optimal combination of control factor levels can be determined.

When the oil yield ratio was analysed, the higher temperatures generated the best responses. Levels 1 and 2 of the granulometry factor (ground and broken seeds) gave the highest oil yield. With respect to factor C, it is suggested that the best response was achieved at the highest exposition times.

In the case of the acidity value, the lower temperatures and 5 min of factor C generated the best responses, and broken and entire seeds were less affected by the hydrothermal pretreatments.

With respect to peroxide index, using the lower temperatures, entire seeds and 15 min of exposition time produced the best response.

Table 5. Signal-noise values associated with oil yield, acidity and peroxide index

	Signal-noise		
Experiment	Oil yield ratio	Acidity value	Peroxide index
E1	41.0	1.8	−12.0
E2	41.4	4.0	−10.7
E3	40.2	3.2	−8.4
E4	40.0	4.2	−6.4
E5	41.9	0.5	−11.4
E6	41.8	3.5	−12.1
E7	41.3	4.2	−10.6
E8	40.3	4.3	−6.9
E9	42.1	0.4	−12.0

Table 6. Average responses (M_{ij}) of parameter levels for the analysis of oil yield ratio

Control factor	Level 1	Level 2	Level 3
B	40.8	41.2	41.4
C	41.0	41.2	41.1
D	41.7	41.5	40.1

Table 7. Average responses (M_{ij}) of parameter levels for the analysis of acidity value

Control factor	Level 1	Level 2	Level 3
B	3.4	3.0	2.3
C	3.2	2.9	2.7
D	0.9	4.0	3.9

Table 8. Average responses (M_{ij}) of parameter levels for the analysis of peroxide index

Control factor	Level 1	Level 2	Level 3
B	−9.7	−9.7	−10.8
C	−10.3	−9.7	−10.1
D	−11.8	−11.1	−7.2

In the case of factor C, in all cases this was the factor that showed the smallest variation in SN ratio among the three levels, suggesting that it was the factor that least affected the responses.

Analysis of Variance

Taguchi suggests using analysis of variance (ANOVA) to study the significance of the experimental factors.[27] This analysis is shown in Tables 9–11 for the different responses with the corresponding contribution of the factors.

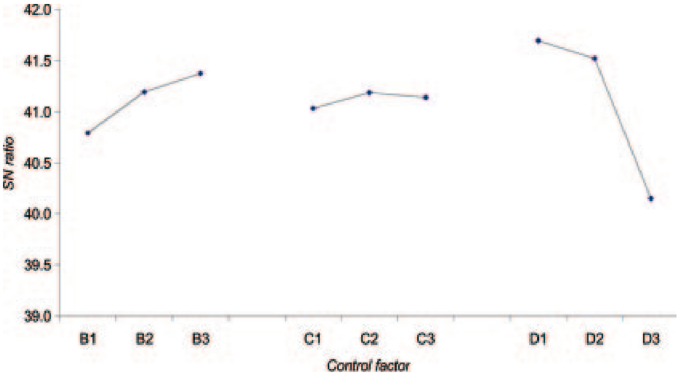

Figure 5. Effect of the control factors when analysing the oil yield ratio response.

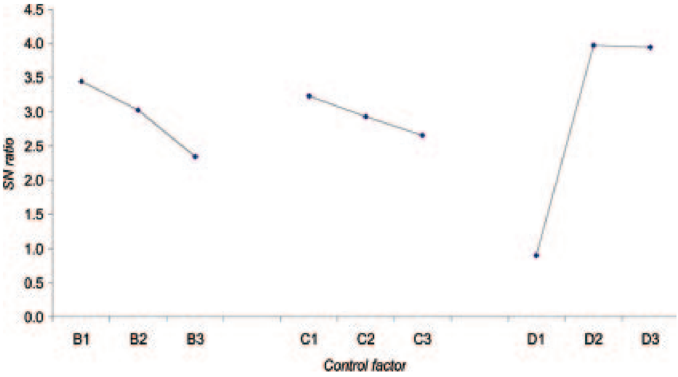

Figure 6. Effect of the control factors when analysing the acidity value response.

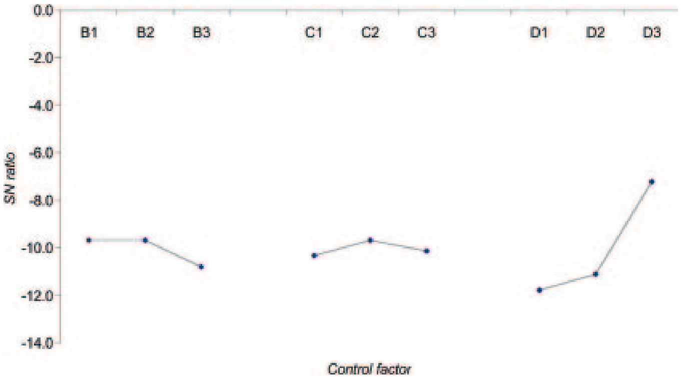

Figure 7. Effect of the control factors when analysing the peroxide index response.

In all cases, a higher contribution of the granulometry factor was observed, and in second place of the temperature factor. The contribution of the time factor was treated as error, confirming the analysis performed above.

Table 12 shows the analysis of differences among factor means (Tukey's test, $P < 0.05$). Tukey's test, complementing the ANOVA, did not reveal significant differences among the three levels of factor C, but it detected differences among the levels of factors B and D. It confirmed what was analysed in Figures 5–7 regarding the optimum values of the factors. At the same time, this analysis also allowed for the detection of possible significant differences among the responses of the variables under study. For example, when analysing oil yield, no significant differences were observed between level 2 and 3 of the temperature variable (B); however, there were significant differences among the three levels of granulometry (D).

Since the analysed quality parameters gave acceptable values for trade standard sin all the experiments, the optimum levels of the factors were then selected taking into account only oil yield whose significant factors were temperature and granulometry (Table 9). The optimum levels were 120 °C and 130 °C for temperature (levels 2 and 3) and granulometry of broken or ground seeds (levels 1 and 2) (Table 2), not considering exposition time. In order to verify the method, it was necessary to select an additional experiment (E10) in which the optimum operating conditions (120 °C, 5 min, broken seeds) were also verified. This last experiment was selected as optimum because it complied with the optimum levels obtained the by Taguchi method (120 °C or 130 °C for the temperature factor; time of 5, 15 or 30 min indistinctively, and ground or broken seeds), and also because it used the shortest tested time, the lowest optimum temperature level and the lesser grinding work of the two optimum granulometries (broken seeds), thereby producing better quality values. As a result, this optimum experiment was the least expensive.

Confirmation Test

The predicted response of an additional experiment ($\mathrm{SN}_{\mathrm{pr}}$) can be calculated using Equation (4), based on the average response M_{ij}, when the three factors are at their lowest level.[11–13]

$$\mathrm{SNpr} = \overline{\mathrm{SN}} + (M\mathrm{B}1 - \overline{\mathrm{SN}}) + (M\mathrm{C}1 - \overline{\mathrm{SN}}) + (M\mathrm{D}1 - \overline{\mathrm{SN}}) + \mathrm{error} \quad (4)$$

The overall mean $\overline{\mathrm{SN}}$ is the average response of all the tests of the proposed experimental design. If the experimental results are close

Table 9. ANOVA. Response analysed: oil yield ratio (%)

Variance sources	Sum-of-squares (SS)	Degree of freedom (DF)	Mean square (MS)	Pure SS (SS′)	Contribution
B	0.54	2	0.27	0.37	7.23%
C	0.04	2	—	—	—
D	4.25	2	2.125	4.09	79.88%
Error	0.29	2	—	—	—
Pooled error	0.33	4	0.0825	0.66	12.89%
Total variance	5.12	8	—	—	100%

Table 10. ANOVA. Response analysed: acidity value (oleic acid %)

Variance sources	Sum-of-squares (SS)	Degree of freedom (DF)	Mean square (MS)	Pure SS (SS′)	Contribution
B	1.79	2	0.895	1.45	6.83%
C	0.47	2	—	—	—
D	18.76	2	9.38	18.43	86.85%
Error	0.20	2	—	—	—
Pooled error	0.67	4	0.1675	1.34	6.31%
Total variance	21.22	8	—	—	100%

Table 11. ANOVA. Response analysed: peroxide index (meq O_2/kg oil)

Variance sources	Sum-of-squares (SS)	Degree of freedom (DF)	Mean square (MS)	Pure SS (SS′)	Contribution
B	2.61	2	1.305	2.06	5.17%
C	0.64	2	—	—	—
D	36.14	2	18.07	35.59	89.33%
Error	0.45	2	—	—	—
Pooled error	1.09	4	0.2725	2.19	5.50%
Total variance	39.84	8	—	—	100%

Table 12. Comparison of means of the different responses

Factor	Level	Oil yield ratio (%)	Acidity value (oleic acid %)	Peroxide index (meq O_2/kg oil)
B	1	109.8a	0.68a	3.16a
	2	115.1b	0.72a	3.10a
	3	117.7b	0.77b	3.53b
C	1	112.9a	0.69a	3.40a
	2	115.2a	0.73a	3.16a
	3	114.5a	0.75a	3.23a
D	1	121.7c	0.90b	3.88b
	2	119.1b	0.63a	3.61b
	3	101.8a	0.63a	2.31a

Different letters for each factor and in the same column indicate significant differences (Tukey's test, $P < 0.05$).

to those predicted by the model, then the "no interaction" assumption should be correct. The approximate error was calculated using the "pooling technique".[9]

In order to confirm the obtained results, an additional experiment was carried out (E10) in which broken seeds were exposed to steam at a temperature of 120 °C for 5 min.

In the analysis of oil yield, it was considered that the contribution of factor C can be treated as error since it had the lowest mean square in ANOVA (Table 9); thus these terms are simplified, and the predicted SN value (SN_{pr}) for E10 is:

$$SN_{pr} = 41.2 + (41.2 - 41.2) + (41.5 - 41.2) = 41.5 \qquad (5)$$

corresponding to an oil yield value equal to 120.2% oil yield ratio, calculated by Equation (1).

Table 13 presents the values of oil yield ratio, acidity and peroxide index calculated using SN_{pr} (Equation (4)) for the conditions of the confirmation experiment (120 °C, 5 min, broken

Table 13. Comparison between the values obtained experimentally and those predicted by the model for the verification test (E10)

	Oil yield ratio (%)	Acidity value (% oleic)	Peroxide index (meq O_2/kg oil)
Verification test	120.2 ± 2.0	0.68 ± 0.06	ND
Predicted value	120.2	0.62	3.7

seeds) and the values of E10 obtained experimentally. For oil yield, the relative error between the predicted value and the observed value was lower than 0.1%, therefore Taguchi's "no interaction" model was in good agreement with experimental results.

A difference of 8% was observed in the acidity between the predicted value and the real value. Thus, experiment E10 would confirm the proposed model, with a slight difference between the results due to the possible existence of interactions among factors.

When the peroxide index response was analysed, peroxides were not detected in the confirmation experiment (E10), whereas the model had predicted a value of 3.7 meq O_2/kg oil. Therefore, in this case the model did not adequately predict the response, and interactions among the analysed factors could not be neglected.

This optimum experiment produced a relative increase in oil yield of 20.2% compared with the values of the untreated sample, whereas the acidity value reached 0.68%, and the amount of peroxides was not affected when comparing the oil obtained from the hydrothermally treated sample with that extracted from untreated seeds.

This species was stable to oxidation during the hydrothermal pretreatments. This stability could be associated with a low polyene index, as well as with the inactivity of oxidative enzymes (lipase, peroxidase, lipoxygenase) due to the hydrothermal treatment,[28] reducing the oxidation rate of the oil.

The quality of the oil extracted from hydrothermally treated seeds was affected to a large extent due to the prior grinding of the seeds. The steam treatment of entire seeds did not cause a significant quality loss in the oil. This behaviour could be explained by the effect of the protective barrier of the seed coat preventing the access of steam and oxygen to the oil. However, the pretreatment did not improve the oil yield, and its incorporation was of little use to increase oil yield in entire seeds. In addition, it has been reported that this pretreatment does improve the dehulling efficiency of entire seeds,[3] and the plasticity necessary in seeds before flaking. This treatment would not affect the quality parameters analysed in this work.

CONCLUSIONS

The "no interactions" model proposed by Taguchi proved to be efficient to analyse the oil yield obtained by solvent extraction from hydrothermally pretreated canola seeds. In the case of the acidity index, the model predicted a value close to the real value. However, the model did not adequately predict the response of peroxide index because the interactions among factors were not taken into account, which should not be neglected when analysing this response.

By using the Taguchi model it was possible to determine the optimum operating conditions and confirm them with an additional experiment in which a significant improvement in oil yield was observed (20%), with no significant effect on the peroxide index. A negative effect was observed on acidity, but its value did not exceed that established by the trading rules of the Canadian Oilseed Processors Association (COPA). The optimum experiment was carried out by steaming the broken seeds at 120 °C for 5 min.

NOMENCLATURE

A, B, C, D	factors used in the experimental design
M_{ij}	average response of factor i at level j
n	number of measurements for each experiment
SN	signal-noise ratio
SN_{pr}	signal-noise predicted by the model
$\overline{SN}$	overall mean signal-noise ratio
x_k	response obtained experimentally

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REFERENCES

[1] A. Szydlowska-Czerniak, G. Karlovits, A. Sosna-Sárdi, C. Dianokzki, *J. Am. Oil Chem. Soc.* **2009**, *86*, 817.

[2] J. Fornal, J. Sadowska, R. Jaroch, T. Winnicki, *Pol. J. Food Nutr. Sci.* **1996**, *5*, 25.

[3] N. Thakor, S. Sokhansanj, I. McGregor, S. McCurdy, *J. Am. Oil Chem. Soc.* **1995**, *72*, 597.

[4] J. Mohamadzadeh, A. Sadeghi, M. Yaghbani, M. Aalami, *World J. Dairy Food Sci.* **2009**, *4*, 14.

[5] K. K. Singh, D. P. Wiesenborn, K. Tostenson, N. Kangas, *Trans. ASAE* **2002**, *45*, 1029.

[6] N. A. M. Eskin, B. E. Mc, R. Donald, L. J. Przybylski, R. Malcolmson, T. Scarth, K. Mag, D. Ward, D. Adolph, *A&G* **2006**, *65*, 610.

[7] M. A. Williams, "Libro de Oro de A&G," Editorial Amalevi, Rosario **2000**, 484.

[8] Y. Liu, W. Chang, Y. Yamagata, *CIRP-JMST* **2010**, *3*, 40.

[9] C. Özdemir, A. N. Akın, R. Yıldırım, *Appl. Catal. A: Gen.* **2004**, *258*, 145.

[10] O. N. Ata, M. Yeşilyurt, S. Çolak, A. V. Beşe, *Can. J. Chem. Eng.* **2002**, *80*, 1.

[11] J. Zhou, D. Wu, D. Guo, *J. Chem. Technol. Biotechnol.* **2010**, *85*, 1402.

[12] A. V. Beşe, N. Borulu, M. Çopur, S. Çolak, O. N. Ata, *Chem. Eng. J.* **2010**, *162*, 718.

[13] T. Havuz, B. Dönmez, C. Çelik, *J. Ind. Eng. Chem.* **2010**, *16*, 355.

[14] A. Rajaei, M. Barzegar, Y. Yamini, *Eur. Food Res. Technol.* **2005**, *220*, 401.

[15] ASAE, "Standards Engineering Practices Data," 46th edition, American Society of Agricultural Engineers, St. Joseph **1999**, p. 557.

[16] AOCS, "Official Methods and Recommended Practices of the American Oil Chemists Society", 5th edition, D. Firestone, Eds., AOCS Press, Champaign **1997**.

[17] IUPAC, "Standard Methods for the Analysis of Oils, Fats and Derivatives," *International Union of Pure and Applied Chemistry*, 7th edition, C. Paquot, A. Hautfenne, Eds., Blackwell Scientific, Oxford **1992**.

[18] M. B. Fernández, E. E. Perez, G. H. Crapiste, S. M. Nolasco, *J. Food Eng.* **2012**, *111*, 682.

[19] N. G. Izquierdo, L. A. N. Aguirrezábal, F. H. Andrade, V. R. Pereyra, *Field Crops Res.* **2002**, *77*, 115.

[20] M. A. Burnet, M. B. Fernández, G. H. Crapiste, S. M. Nolasco, E. E. Perez, *A&G* **2012**, *88*, 480.

[21] D. C. Montgomery, *Diseño y Análisis de experimentos*, Editorial Grupo Editorial Iberoamérica, México **1991**, p. 369.

[22] R. Romero, L. R. Zúnica, “XIX Congreso Nacional de Estadística e Investigación Operativa”, Universidad Nacional de Educación a Distancia, Alicante **1991**, p. 92.

[23] F. B. Padley, F. D. Gunstone, J. L. Harwood, “Occurrence and Characteristics of Oils and Fat”, *The Lipid Handbook*, 2nd edition, F. D. Gunstone, J. L. Harwood, F. B. Padley, Eds., Chapman & Hall, London **1994**, p. 89.

[24] L. Iriarte, O. Valetti, *A&G* **2006**, *65*, 646.

[25] L. Debonte, D. Iassonova, L. Liu, W. Loh, *Lipid Technology* **2012**, *24*, 175.

[26] P. Zacchi, R. Eggers, *Eur. J. Lipid Sci. Technol.* **2008**, *110*, 111.

[27] M. S. Phadke, “Quality Engineering Using Robust Design,” Prentice Hall, Englewood Cliffs, NJ **1989**, p. 51.

[28] A. Szydlowska-Czerniak, G. Karlovits, G. Hellner, C. Dianokzki, *Process Biochem.* **2010**, *45*, 247.

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