

# Symposium

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## Scale-Up of Canola Oil Degumming

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### ABSTRACT

The chemical degumming of canola oil was optimized using citric acid and maleic anhydride as degumming agents. These chemicals were selected from a group of 54 degumming agents, reported previously. The effect of temperature, chemical addition level, water addition level and contact times was investigated. Best results were obtained at 40 C, using 10 min contact with the chemical, followed by the addition of 2% water and agitation for 20 min.

Chemical degumming reduced the residual phosphorus level from 1049 mg/kg to 50 mg/kg using either maleic anhydride or citric acid. Refining tests gave excellent deodorized or hydrogenated products.

The optimized reaction conditions were applied to 330 kg test batches of oil in the P.O.S. Pilot Plant. Results were identical to those obtained in the laboratory, indicating that the process may be scaled up readily for industrial application.

### INTRODUCTION

In 1978, the Rapeseed Association of Canada, now the Canola Council of Canada, commissioned a study of chemical degumming techniques for crude rapeseed oil produced from the new, canola, varieties of rapeseed. These varieties contain low levels of erucic acid and glucosinolates. The initial phase of the work identified 4 chemical degumming agents that met the project goals of 100 mg/kg residual phosphorus in the degummed oil. These compounds were phosphoric, nitric and citric acids and maleic anhydride (1).

Based on the results of laboratory refining tests, maleic anhydride and citric acid were selected for further study. The following report summarizes the results of laboratory optimization and pilot plant tests, which were presented at the 1982 AOCS Annual Meeting in Toronto (2).

### EXPERIMENTAL

#### Materials

Laboratory optimization studies were carried out using a

single batch of extracted candle oil prepared by the P.O.S. Pilot Plant Corporation, University of Saskatchewan Campus, Saskatoon, Saskatchewan. Pilot plant tests were performed on a 5000-lb batch of commercial canola oil, which was a blend of prepressed and extracted oils. The oils contained 1078 and 541 mg/kg phosphorus, respectively.

Food-grade citric acid and reagent-grade maleic anhydride was used in all tests. Selected degummed oil samples were refined in the laboratory, using reagent-grade NaOH and Pembina acid-activated bleaching clay (Pembina Mountain Clay Co. Ltd., Winnipeg, Manitoba).

#### Degumming Technique

The laboratory degumming technique described earlier was used (1). 300 g of oil were preheated to the reaction temperature, with agitation. Citric acid was added as 50% aqueous solution, and maleic anhydride was added as a powder. This mixture was agitated for a measured time period, then 2% water was added, and the agitation continued for a second time period. The gums were removed by filtration, and the degummed oil was analyzed.

The effect of degumming temperature on the residual phosphorus level was examined in the temperature range of 20-80 C. The effect of the level of chemicals added was investigated at 40 C. Starting from 3000 mg/kg, the addition level was decreased in 500 mg/kg steps until a significant change was observed in the residual phosphorus level. As 2500 and 3000 mg/kg gave similar results, higher addition levels were not attempted.

Both the initial mixing time with the degumming chemical, and the subsequent contact time with water were varied in 10-min steps from 10 to 30 min.

Finally, the effect of water addition level was determined using the most effective temperature, concentration and contact time values. One, 2 or 3% water was added to the sample after 10 min of contact with either citric acid or maleic anhydride followed by a further 20 min of agitation.

## CANOLA OIL DEGUMMING

The optimal laboratory degumming procedures for each chemical were scaled up by a factor of 10: the degumming tests were repeated in the laboratory using 3000-g batches of oil.

The tests were repeated in industrial equipment, using 330-kg batches of oil. The facilities of the P.O.S. Pilot Plant Corporation in Saskatoon, Saskatchewan, Canada were used for these tests. The pilot plant equipment for this test consisted of a series of reactor vessels, pumps and centrifuges connected together at a central "patchpanel". All connections were stainless steel. The following equipment was used: (a) 2600 L jacketted reactor, 150 cm I.D., SS 316 Hayward Gordon Model #1-HTA-1.5 agitator, 6 blades with 45° pitch, 50 cm diameter, 125 rpm, (b) 700 L mixing tank, 82.5 cm O.D. 16GA, SS 316 Greyc Model ND-1 VM agitator, 24 cm HI-FLOW propeller, 1/4 HP motor, (c) Westfalia Model SA-7-01-576 automatic desludging centrifuge, 69 discs used, 4 classifier discs at locations 1, 23, 46 and 69, top diameter of disc 6.4 cm bottom diameter 16.2 cm, conical half angle 35°; bowl diameter 24.9 cm, bowl speed 8400 rpm, and (d) 700 L vertical dryer, 90 cm I.D. Hayward Gordon medel VLA-7.5 LS agitator, 4 blades with 45° pitch, 30 cm blade diameter.

In the pilot plant tests, the starting oil was charged to a 2600 liter jacketted reactor for preheating. The oil was heated to 40 C with agitation, then 330 L of the preheated oil were measured and pumped to a 700-L mixing tank for degumming.

Citric acid was added to the mixing tank as a 50% aqueous solution. Maleic anhydride was crushed to a fine powder, then dispersed in the oil directly. With either degumming agent, the oil was allowed to react for 10 min, then water was added (2% by weight of oil), and the mixture was agitated for 20 min.

The mixture was then pumped to a continuous centrifuge for separation of coagulated gums. The centrifuged oil was charged to a batch dryer and was dried for 1 hr at 60-70 C, under 27" Hg vacuum.

In addition to the chemical degumming runs, one standard batch degumming test was also performed as a control. In this run, the oil was heated to 70 C, 2% water was added and the mixture was agitated for 20 min. The resulting oil was centrifuged and dried as before. Centrifuge throughputs were 2-3kg/1 min, and the desludging interval was 10 min.

Samples of each degummed oil were alkali refined in the laboratory using the method described previously (1).

## DISCUSSION OF RESULTS

The laboratory experiments demonstrated that the residual phosphorus concentration in the degummed oil varies significantly with temperature. The highest phosphorus levels were observed at 80 C, which is near the usual industrial degumming temperatures. As illustrated in Figure 1, the best results were obtained at 40 C. At lower temperatures, the dispersion and reaction of the degumming agents was hampered by the increased viscosity of the oil, whereas, in our experience, at higher temperatures the hydratable phospholipids are more soluble in the oil, resulting in higher residual phosphorus levels.

The residual phosphorus level increased as the concentration of the degumming agent was decreased below a threshold level. In the case of citric acid, addition levels of 2500 ppm or more resulted in phosphorus levels of ca. 40 ppm. The phosphorus concentration increased rapidly as the citric acid concentration was reduced below 2000 ppm. With maleic anhydride degumming, addition levels between 1500 and 3000 ppm did not affect the phosphorus content

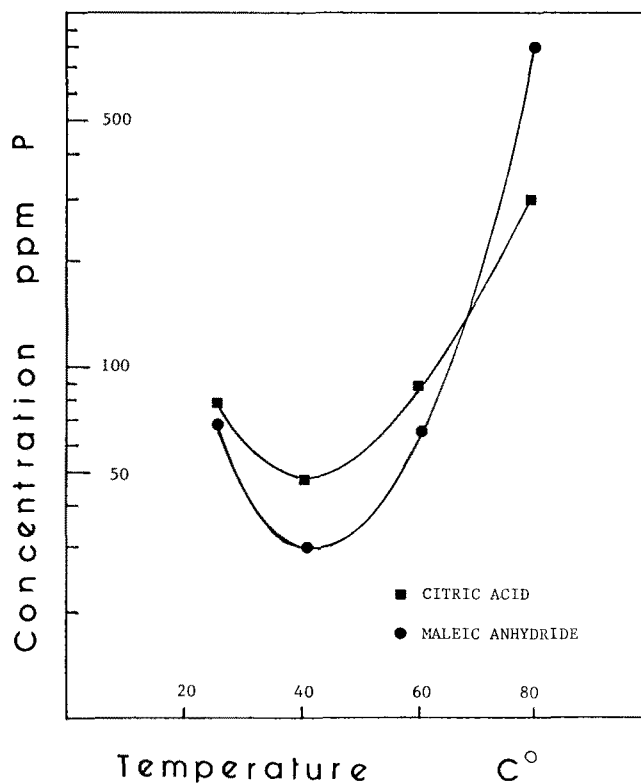


FIG. 1. Variation of residual phosphorus concentration with degumming temperature. 2500 ppm citric acid or maleic anhydride and 2% water added.

of the degummed oil significantly, but lower levels resulted in an exponential increase in phosphorus concentration. The results are presented in Table I. In further tests, citric acid concentration of 2500 ppm and maleic anhydride concentrations of 1500 ppm were used.

Following the optimization tests on degumming temperature and chemical addition levels, both the chemical contact time and the subsequent contact time with water were examined using the established temperature and chemical addition levels. The contact times were varied from 10 to 30 min in 10-min stages. The results are presented in Table II.

The 2 chemicals behaved similarly, despite the fact that citric acid was added to the oil as a 50% aqueous solution, whereas maleic anhydride was added as a powder. Chemical contact times did not have a significant effect. The optimum water contact time matched the preliminary screening test conditions of 20 min final agitation, confirming our initial estimates which were based on previous experience with rapeseed oil refining.

TABLE I

Effect of Concentration on Residual Phosphorus<sup>a</sup>

Addition level	Citric acid Degumming	Maleic anhydride Degumming
mg/kg	Residual phosphorus mg/kg	
500	—	194
1000	—	65.5
1500	139	29.3
2000	74	29.2
2500	38.6	27.6
3000	37.6	27.6

<sup>a</sup>Reaction conditions: 40 C, 10 min contact time, 2% water added with 20 min further contact.

**TABLE II**  
Effect of Contact Time

Contact time with chemical min	Contact time with water min	Residual phosphorus	
		Citric acid degummed oil mg/kg	Maleic anhydride degummed oil mg/kg
30	30	38.2	33.5
20	20	38.6	33.6
10	20	38.7	33.9
20	10	70.0	89.1
10	10	91.4	120.6

Three water addition levels were examined. Three percent water did not produce a significant improvement over the results obtained with 2% water with either reagent. The reduction of the water addition level to 1% approximately tripled the residual phosphorus levels, indicating that 2% water is required for adequate degumming.

Larger scale laboratory tests, using 3000-g batches of oil, confirmed that both maleic anhydride and citric acid will produce excellent degummed oils with less than 50 ppm phosphorus content at the optimum conditions defined by the small-scale laboratory tests.

#### Pilot Plant Tests

Duplicate tests were performed on 330-kg batches of oil with each reagent and the results were compared with those of a conventional water degumming run. The results are summarized in Table III.

The results demonstrate that chemical degumming, with either citric acid or maleic anhydride as the degumming agent, will drastically reduce the residual phosphorus levels in degummed "Candle" oil.

The consistency of the heavier, gum phase remained constant in the 2 citric acid runs. It is suspected that the difference in the residual phosphorus levels between the runs was caused by differences in the time required to achieve the necessary dispersion of citric acid. This was probably a result of the "batch" addition of the citric acid solution to the oil, which could be overcome easily in an industrial application through the use of inline mixing.

In the maleic anhydride runs, the deviation in the residual phosphorus level was smaller than that of the citric acid runs. Although maleic anhydride seemed to coagulate phospholipids better than citric acid, it caused a buildup of gums within the bowl of the centrifuge. The coagulation effect results in great variations in gum consistency, and it may have caused the difference of the residual phosphorus levels in the degummed oils. It is difficult to predict whether this would be a problem in an industrial application.

In all runs, the neutral oil content of the gums was

similar. Maleic anhydride gave slightly lower values of  $36\pm 1\%$  compared with  $41\pm 1\%$  for citric acid.

The degummed oils produced by runs No. 1, 3 and 5 were refined, bleached and deodorized in the laboratory. The results of the refining tests are summarized in Table IV. The addition of phosphoric acid was eliminated for both of the chemically degummed oils.

The laboratory and pilot plant tests were fully consistent and showed that canola oil may be degummed chemically to produce degummed oils with residual phosphorus levels well below 100 ppm. Citric acid and maleic anhydride were especially successful in producing high quality degummed oils that resulted in excellent refined, bleached, deodorized and hydrogenated oils with satisfactory flavor, color and stability.

Both citric acid and maleic anhydride are readily available at comparable prices. Although the required addition level for maleic anhydride is substantially lower than that of citric acid, this material is difficult to handle. It is a strong irritant and may be toxic in high concentrations. Thus, it may not be practical to apply the maleic anhydride degumming process in existing crushing plants, and the use of the gums may be limited.

On the other hand, citric acid is a well known food additive that is regularly used in edible oil refineries. It acts as a chelating agent, and, therefore, its presence in the gums, if added back into the meal, will improve its stability. Accordingly, the citric acid degumming technique was determined to be the most suitable process of chemical degumming for Canadian rapeseed crushing plants.

Conventional steam degumming plants may be converted readily to the citric acid degumming process presented here. The process consists of two contact stages: an initial contact with the aqueous solution of citric acid, followed by water contact. The subsequent separation and drying stages are identical to the current industrial practice. Thus, an existing degumming system may be modified by the addition of two tanks, agitators, appropriate pumps and temperature-controlling equipment.

Chemical degumming offers a number of potential advantages: (a) since a larger proportion of the phospholipids are recovered in the crushing plant, these may be added back to the meal for an economic return, (b) the phosphoric acid pretreatment step may be eliminated from the conventional alkali refining process, (c) refining losses due to the emulsifying action of phospholipids, and the resulting entrainment could be reduced, (d) the acidulated soapstock will be easier to split due to the lower emulsifier content, (e) the waste load from the refinery will be reduced due to both lower neutral oil losses and the reduction in the amount of gums discharged and (f) the oil is more suited to physical refining techniques due to the significant reduction in nonvolatile contaminants.

Since the process behaved identically at scales ranging

**TABLE III**  
Results of Pilot Tests

	FFA %	P mg/kg	Fe mg/kg	Ca mg/kg	S mg/kg
Starting	0.61	541	2.5	224	3.2
Water degummed	0.61	185	1.24	172	1.9
Citric acid degummed	0.54	64	0.91	50.8	2.0
Maleic anhydride degummed	0.53	50	0.68	40.4	1.6
	0.46	41	0.96	26.8	1.8
	0.47	33	0.97	20.3	1.6

## CANOLA OIL DEGUMMING

TABLE IV

## Results of Refining Tests

Oil sample		Color		FFA %	PV mg/kg	IV	P mg/kg	Fe mg/kg	Ca mg/kg	S mg/kg	AOM mg/kg
		R	Y								
Water degumming											
Starting oil	(1")	3.5	50	0.70	4.62	120.8	541	2.47	224	3.2	—
Degummed oil	(1")	3.2	40	0.61	5.82	121.2	185	1.24	172	1.9	—
Neutralized oil	(1")	2.7	35	0.03	—	—	0.40	0.55	5.1	—	—
Bleached oil	(1")	0.2	1.5	0.07	—	121.5	0.31	0.25	2.3	—	—
Deodorized oil	(5 1/4")	0.4	4	0.02	0.0	120.8	—	—	—	0.21	20
after 2 weeks	(5 1/4")	0.5	5	0.02	0.41	—	—	—	—	—	—
Citric acid degumming											
Starting oil	(1")	3.5	50	0.70	4.62	120.8	541	2.47	224	3.2	—
Degummed oil	(1")	3.1	40	0.53	6.20	121.8	50.0	0.68	40.4	1.6	—
Neutralized oil	(1")	2.6	35	0.03	—	—	8.2	0.50	17.6	—	—
Bleached oil	(1")	0.2	1.5	0.07	—	121.6	0.38	0.36	1.7	—	—
Deodorized oil	(5 1/4")	0.4	4	0.01	0.0	121.3	—	—	—	0.19	20
After 2 weeks	(5 1/4")	0.5	5	0.01	0.38	—	—	—	—	—	—
Maleic anhydride degumming											
Starting oil	(1")	3.5	50	0.70	4.62	120.8	541	2.47	224	3.2	—
Degummed oil	(1")	3.1	35	0.46	5.64	121.2	33.3	0.96	20.3	1.5	—
Neutralized oil	(1")	2.4	35	0.03	—	—	0.73	0.39	2.9	—	—
Bleached oil	(1")	0.2	1.5	0.06	—	120.8	0.15	0.38	1.5	—	—
Deodorized oil	(5 1/4")	0.3	3	0.01	0.0	120.5	—	—	—	0.25	20
after 2 weeks	(5 1/4")	0.4	4	0.01	0.39	—	—	—	—	—	—

from 300 g to 330 L, a scale-up factor of ca. 1,000, we feel confident that the results may be reproduced easily in a commercial operation. Further product improvement may be expected from more uniform contact in a fully continuous process.

## ACKNOWLEDGMENTS

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## Current Practices in Continuous Cottonseed Miscella Refining

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## ABSTRACT

Miscella refining of crude cottonseed oil has become a generally accepted commercial process for the past 20 years. The simple and efficient continuous process for removal of undesirable impurities is described, having changed little in its basic form since discovery 40 years ago. The individual unit processes, control systems, process flow charts, chemical reactions and oil-to-hexane ratios used in miscella refining are described. The several advantages to miscella refining vs conventional oil refining are noted.

## INTRODUCTION

The solvent extraction of crude oil from cottonseed has been a commercial reality for the past 60 years. Before solvent extraction, the crude cottonseed oil was extracted mechanically by hydraulic and expeller-type presses. The mechanically extracted crude oils typically contain fewer nonglyceride impurities than solvent-extracted oils, making them easier to refine, but having the disadvantage of leaving

a higher oil content in the meal. After World War II, the prepressing of cottonseed, followed by solvent extraction and direct solvent extraction, has become increasingly predominant in the industry. Solvent-extracted cottonseed has resulted in high oil yields, but also higher oil refining losses. The higher refining losses are due to the greater extraction of gossypol and other nonglyceride components and a decrease in the oil content of the solvent extracted meal to 1% or less (1).

The high-temperature solvent stripping following extraction yields a very dark colored crude oil. The dark color in the crude oil may be difficult to remove by conventional refining methods, if the color has been set by high-temperature degradation products of gossypol and other related pigments (1).

Of the many hydrocarbon solvents investigated for use in oilseed extraction, commercial hexane has become the most widely used solvent. With hexane as the dominant