Sulfur Content of Crude Rapeseed Oil from Aqueous Extraction

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ABSTRACT

Dry heating of rapeseed before grinding increased the sulphur content of the crude rapeseed oil. Boiling the seeds in H_2O before grinding caused a slight decrease in the sulphur level of the oil. Control of pH during boiling of the ground rapeseed increased the sulphur content of the oil from ca. 2 ppm at pH 3.1 to ca. 10 ppm at pH 7.6.

INTRODUCTION

Studies on the feasibility of aqueous extraction of rapeseed oil by a laboratory procedure and the quality of the crude oil obtained (AO) have been reported (1). This paper gives further details on sulphur content of crude rapeseed oils as affected by pretreatments for the aqueous extraction procedure.

MATERIALS AND METHODS

All experimental or industrial materials, the laboratory extraction procedures, and the analytical methods used were the same as previously described (1). The three rapeseed varieties available for this study (*Brassica campestris*L., var. Echo, Span, and Torch) contained 5.6, 9.5, and 6.4% moisture, and 42.6, 46.2, and 46.3% oil, respectively. The Echo seeds had been stored at 5 C for 5 yr before the experiments; consequently, their viability was found to be about 45%. The viability of the Span and Torch materials was ca. 99% as determined by germination tests.

RESULTS AND DISCUSSION

Sulfur Contents of the Laboratory Crude Oils

The sulfur contents of the AO, soxhlet, and industrial crude oils are shown in Table I. The sulfur content of the AO from untreated seeds of the Echo variety was 4.5 ppm and that from untreated seeds of the Span variety was 6 ppm. These values were higher than that of the soxhlet extracted crude oils (less than 2 ppm) but lower than that of the industrial crude oil (10 ppm). Typical sulfur contents in Canadian industrial crude oil were reported as 16-57 ppm (2,3). Low levels of sulfur (0-5 ppm) were also obtained by other workers in laboratory extracted oils of untreated seeds (3,4). One of the initial steps of the aqueous extraction of the rapeseed oil was the boiling of the ground rapeseed (1). The inactivation of myrosinase by boiling prevents the hydrolysis of glucosinolates into sulfur-containing compounds which are soluble in oil, thereby resulting in an oil with lower sulfur content. If the seed was boiled before grinding, the sulfur levels of the oils were 3 ppm for the Echo variety and 4 ppm for the Span variety.

The Effect of Dry Heating

Heating the seed before grinding influenced the sulfur level of the experimental oil. Dry heat treatment of rapeseed before grinding was carried out in order to reduce the moisture content of the seed so that the grinding operation would be faster and the rapeseed particles would be less sticky. However, the results show that heating the rapeseed

TABLE I

Sulfur Content of Crude Rapeseed Oils

Sample and treatment	Sulfur content (ppm)[a]
A. Industrial crude oil	
B. campestris L. var. Span	10
B. *B. campestris* L. var. Echo	
1. Aqueous extration	
a) Untreated seed	4.5
b) Seed boiled	3
c) Seed dried in oven at 105 C for 2 days	23
2. Soxhlet extraction (untreated seed)	2
C. *B. campestris* L. var. Span	
1. Aqueous extraction	
a) Untreated seed	6
b) Seed dried at 55 C for ca. 17 hr.	15
c) Seed dried at 105 C for ca. 17 hr.	12
d) Seed boiled and dried at 105 C for ca. 17 hr.	4
D. *B. campestris* L. var. Torch	
1. Aqueous extraction (seed dried at 55 C for 3 days)	50
2. Soxhlet extration (untreated seed)	2

[a]Average of two analyses. All samples under B, C and D were composite samples of at least five extractions each.

before grinding increased the sulfur content of the oil (Table I). When Span seed was heated at 55 and 105 C for ca. 17 hr, the levels of sulfur in the oils were 15 and 12 ppm respectively. The sulfur content of oil from the Echo seed, heated at 60 C for 2 days, was 23 ppm. Higher sulfur content (50 ppm) was found in oil extracted from Torch seed which was heated at 55 C for 3 days. These results indicate that the level of sulfur in the oil increased with the heating time. Increase in sulfur content of the oil due to the heating of rapeseed was also reported by other workers (3,5). This increase of sulfur in the oil is likely due to the

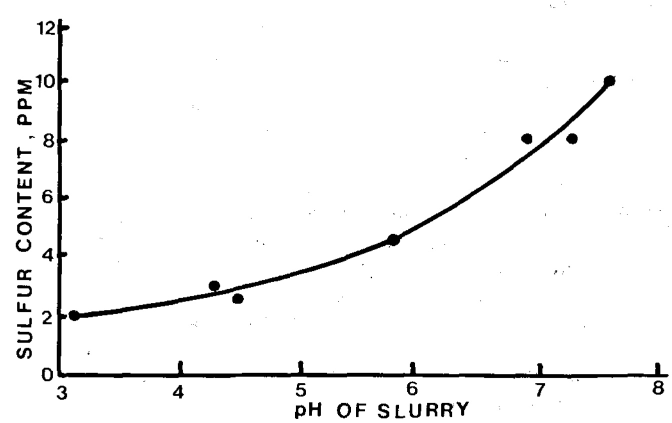

FIG. 1. Effect of pH of rapeseed slurry during boiling on the sulfur content of the oil.

hydrolysis of glucosinolates and not to the partial pyrolysis of glucosinolates (3,5) since heating of the boiled rapeseed did not increase the sulfur level of the oil.

The Effect of pH

The pH during boiling of the ground seed (1) had a significant effect on the sulfur level in the oil (Fig. 1). The sulfur content of the oil increased with increasing pH from ca. 2 ppm at pH 3.1 to ca. 10 ppm at pH 7.6. This could be due to the fact that when the ground rapeseed in the form of clumps was added to the boiling water, inactivation of myrosinase was not instantaneous, especially in the core of the clumps. Before the enzyme inactivation, hydrolysis of the glucosinolates could occur; the extent of this hydrolysis appears to be pH dependent as shown previously by Sosulski et al. (6).

The effect of pH during the subsequent stirring of the boiled ground rapeseed (1) was also examined. However, pH adjustments between 3.9 and 9.7 caused no variation in the sulfur content of the oil.

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The Effect of Processing Operations on the Total Sulphur Content in Rapeseed Oil

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ABSTRACT

The technological difficulties due to the presence of sulphur-containing compounds in rapeseed oil lead to the problem of proper hydrogenation under commercial conditions. The effect of refining operations on the total sulphur content of rapeseed oil was studied. Degumming (phosphoric acid treatment) had an insignificant effect on the sulphur content while deacidification (alkali refining) removed about 30% and bleaching with Fuller's earth removed about 50% of the total sulphur in commercial crude oil. Deodorization affected nearly the complete removal of residual sulphur left after the application of treatments mentioned above.

INTRODUCTION

Today rapeseed ranks fifth among the major oilseeds of the world and is on top in Pakistan. Rapeseed oil, the bulk of which is produced by expression, particularly in developing countries, is mainly processed in the same way as other vegetable oils. The main aim of processing or refining is to remove impurities that contribute unpleasant flavor, which in case of rapeseed oil is chiefly due to sulphur compounds. The level of sulphur compounds (isothiocyanates) in crude rapeseed oil depends on the species, pre-extraction treatment of the seed, and the method of extraction (1-4).

Besides being responsible for undesirable flavors, the presence of sulphur compounds is not only nutritionally objectionable but also causes severe catalyst poisoning during the process of hydrogenation (5-7). The other natural impurities that need elimination to the maximum are free fatty acids, mono- and diglycerides, phosphatides, pigments, proteins and various oxidation products. The processing of rapeseed oil normally consists of degumming, deacidification, bleaching and deodorization. The influence of these unit operations on the total sulphur content of rapeseed oil was studied under industrial conditions.

MATERIAL AND METHODS

Commercially available crude rapeseed oil was analyzed for various physical and chemical characteristics (8) and subjected to processing under industrial conditions at the local processing plant of Morafco Industries Ltd., as given below.

Degumming. The crude oil heated to 70 C in the preneutralizer was treated with 4000 ppm of food grade, 85% phosphoric acid, mixed for 30 min by slow speed mechanical mixer, allowed to settle, centrifuged and finally washed with water at 40 C.

Deacidification. The degummed oil was sprayed over with 15Bé alkali (NaOH – caustic soda) solution at 45 C. The amount of alkali varied according to the free fatty acid content of the oil lot. After contact time of 15 min the mixture was heated to 70 C to facilitate the "break" of emulsion. The soap-stock was separated after settling for 45 min. The neutralized oil so obtained was washed with water to eliminate traces of soap.

Bleaching. The deacidified oil was charged into a bleaching vessel and heated to 90 C under vacuum to ensure complete drying. A mixture of acid-treated Fullers' earth and activated charcoal (20:1) at ca. 3% of the oil volume was added at 85 C and mixed by mechanical agitation for 15 min. Then the oil was cooled to 70 C and filtered through filter press.

Deodorization. The bleached oil was then deodorized under vacuum in steam-stripping conditions (200 C, 6 mm pressure) for 8 hr to remove odor volatiles present in the oil. The deodorized oil was cooled to 50 C before discharging it to storage tanks.

In the first run, ten different batches (each 2 to 2.5 tons) of oil were processed per detail given above, and the sulphur content of the oil in the form of allylisothiocyanate was estimated according to the method