MEHL (EIWEISS) / MEAL (PROTEIN) / TOURTEAU (PROTEINE)

DETOXIFICATION OF RAPESEED MEAL

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Introduction

Recent work at our laboratory has shown that freshwater fish, such as carps, can be grown successfully on feeds containing as much as 40 % of full-fat ground rapeseeds of conventional varieties. Full-fat rapeseed meal is now being used to some extent in fish-farming.

Although ground rapeseed without any further treatment seems to be useful as fish feed, its direct use in food and in the feed for cattle, swine and poultry is limited, largely due to the presence of glucosinolates in the seed.

Rapeseed can be processed to yield two types of meals having different fat content. A <u>defatted meal</u> can be produced by milling and hexane extraction, whereas a <u>full-fat meal</u> can be prepared simply by dehusking and grinding. For use in nutrition, both types of rapeseed meal must be "detoxified" by removing the glucosinolates.

Being water-soluble, intact glucosinolates are easily removed from both defatted rapeseed meal and full-fat ground rapeseed by extraction with water (EAPEN et al., 1969). However, aqueous extraction also removes substantial amounts of other water-soluble constituents of the seeds, which are predominantly proteins. These meal solids extracted together with glucosinolates are lost in the process since it is difficult to recover them from the aqueous extract. Thus, full-fat ground seeds of Brassica napus lose about 20 % meal solids upon extraction with water.

Although a good deal of work has been done towards detoxification of rapeseeds for the production of defatted meal, very little attention has been given to the preparation of high-fat meal.

For the preparation of high-fat protein concentrates, the glucosinolates have been extracted from full-fat ground rapeseed with 30 % aqueous methanol containing 5 % sodium chloride (EKLUND et al., 1971). However, even in this process considerable amounts of meal solids are extracted along with the glucosinolates, apparently due to the high water content of the solvent.

It has been shown that defatted meals from rapeseed and crambe seed can be detoxified by extraction with aqueous mixtures of methanol, ethanol, or acetone (Van ETTEN et al., 1965; RUTKOWSKI, 1970). Some of these solvents apparently remove the glucosinolates without extracting much of the meal solids. Bearing this in mind, we attempted to detoxify full-fat ground rapeseed by extraction with aqueous organic solvents, with an aim to obtain a high-fat meal in good yield. Our basic approach was to find a solvent system which would extract most of the glucosinolates but remove none or very little of lipids and proteins. We studied only those solvents and solvent mixtures which we considered suitable for extraction on an industrial scale.

Experimental

A conventional variety of rapeseed, Brassica napus, was used as starting material. The whole seeds were immersed in boiling water for 2 minutes in order to deactivate the myrosinase (EAPEN et al., 1968). The seeds were then dried to about 5 % moisture level and stored in bulk. This material was used throughout.

In the first series of experiments, extractions were carried out on defatted rapeseed meal. The seeds were ground in a disc mill and defatted with hexane in a Soxhlet apparatus. The defatted meal was desolventized at ca. 40° C and ground to 20-30 mesh. Lots of 10 g of the defatted meal were extracted with 20 g portions of various organic solvents or their aqueous mixtures at room temperature by vigorous stirring for 15 minutes. The mixture was then centrifuged in order to separate the liquid phase. The extracted meal was desolventized, dried and weighed. The loss in weight corresponded to the total amount of material extracted, which consisted mainly of meal solids and little residual lipids. The starting material and the extracted meals were analyzed for isothiocyanates and oxazolidinethione (APPELQVIST and JOSEFSSON, 1967), in order to determine the proportion of glucosinolates removed.

In a second series of experiments, the extraction of full-fat ground rape-seed with pure acetone and aqueous mixtures containing 88.5 %, 80 %, 70 % and 50 % acetone was studied. Rapeseed was ground to a course meal (15-20 mesh) and 50 g lots of this full-fat meal were extracted with 100 g portions of the solvent by vigorous stirring for 30 minutes at room temperature. At the end of the extraction, the solution was drained through a 20 mesh sieve. The extracted meal was desolventized, dried and analyzed for isothiocyanates and oxazolidinethione (APPELQVIST and JOSEFSSON, 1967). The acetone extract was concentrated to remove most of the solvent. The aqueous residue containing lipids and meal solids was then extracted with petroleum ether in order to determine the lipids that had been removed by extraction with acetone or aqueous acetone. Finally, the aqueous residue was dried and weighed in order to determine the amount of meal solids that were extracted.

In a third series of experiments, multiple batch extractions of full-fat ground rapeseed were carried out on a bench scale in order to assess the technical feasibility of the detoxification process. Portions of $500~{\rm g}$ of

ground rapeseed were extracted four times using an equal amount of 70 % aqueous acetone, at each stage. Each extraction was carried out at 40° C by vigorous agitation for 15 minutes. At each stage, the extracted meal and the acetone extract were analyzed as in the preceding experiment. Similarly, a detoxified high-fat meal was prepared by four successive extractions of ground rapeseed with 70 % aqueous acetone using a meal to solvent ratio of 1:2 (instead of 1:1), at each stage.

Results and discussion

In the first series of experiments, the extraction of defatted rapeseed meal with isopropanol, ethanol, acetone, or their aqueous mixtures showed that these solvents removed the glucosinolates and meal solids to varying extent. As expected, increasing water content of the solvent enhanced the extraction of both glucosinolates and meal solids. Among the solvents examined, aqueous acetone was found most effective for the removal of glucosinolates from defatted meal. Using this solvent, as much as 40 % of the glucosinolates were removed by a single extraction at room temperature.

In the second series of experiments, the results were similar to those obtained with defatted meal. Under the conditions used, pure acetone extracted only about 10 % of the glucosinolates but over 60 % of the lipids and practically none of the meal solids. The proportion of glucosinolates extracted increased rapidly with increasing water content of the solvent. As expected, increasing dilution of acetone also resulted in a large reduction in the proportion of lipids extracted and some increase in the amount of extracted meal solids. Thus, under identical conditions, 88.5 % aqueous acetone extracted about 17 % glucosinolates, 2.7 % lipids, and 0.5 % meal solids, whereas 50 % aqueous acetone removed as much as 67 % glucosinolates, 0.4 % lipids, and 6 % meal solids. Further dilution of acetone would have improved the removal of glucosinolates and minimized the extraction of lipids still further, however, the loss of meal solids would have been increased.

We consider 70 % aqueous acetone as the optimum mixture, which ensures sufficient removal of glucosinolates at a minimum loss of lipids and meal solids.

In the third series of experiments, it was found that with successive extractions, the proportions of glucosinolates, lipids, and meal solids removed decreased progressively. After four extractions as much as 90 % glucosinolates were removed. At this stage, a total of about 13 % of the starting material was extracted, 4.2 % as lipids and 8.8 % as meal solids. Thus, a high-fat rapeseed meal containing only 10 % of the original glucosinolates was obtained in a yield of about 87 %.

Apparently, extraction with aqueous acetone removes considerably smaller amounts of meal solids than extraction with water. The total amount

of lipids removed by four extractions with 70 % aqueous acetone corresponded to about 10 % of lipids originally present in the seed. These lipids are easily recovered by concentrating the acetone extract followed by centrifugal separation of lipids from the aqueous phase.

Table 1: Analysis of Rapeseed and Detoxified High-Fat Meal

Sample	Lipids (%)	Isothiocyanates (mg/g)	Oxazolidinethione (mg/g)
Rapeseed (Starting material)	41.2	1.88	5.27
Detoxified high-fat meal x)	37.9	0.14	0.47

x) Ground rapeseed extracted four times for 15 min, each, with 70 % aqueous acetone at 40° C using a meal to solvent ratio of 1;2, at each stage.

Table 1 shows the contents of lipids, isothiocyanates and oxazolindethione of rapeseed and of detoxified high-fat meal, that had been prepared by four successive extractions with 70 % aqueous acetone using a meal to solvent ratio of 1:2, at each stage. It is evident that the detoxified rapeseed meal contains nearly the same level of lipids as the starting material.

These studies show that it might be technically possible to produce detoxified high-fat meal by extraction of rapeseed with aqueous acetone. Though the use of acetone involves additional processing steps, such as desolventizing and solvent recovery, a high yield of detoxified meal might justify the higher processing cost.

We have found that defatted meal too can be detoxified by extraction with aqueous acetone. However, the entire process from seed to detoxified meal would involve two separate extractions using entirely different solvents, namely, hexane for oil recovery, and, aqueous acetone for detoxification. Such a process seems to be impractical, since each of the two solvents has to be recovered separately. We are considering the possibility of oil extraction using concentrated acetone in a similar manner as in a commercial process (VACCARINO, 1961), followed by detoxification with the aid of aqueous acetone. Further work along these lines and on the composition as well as the nutritional properties of detoxified rapeseed meal, rapeseed oil prepared therefrom and protein isolates is in progress in our laboratory.

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