Short Communication

⁂Mass Balance of Hexane Losses in an Extraction Plant

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ABSTRACT

The hexane losses of an extraction plant processing rapeseed have been surveyed over a period of 5 months. The total average loss was 2.0 L/ton of seed according to inventory. By analyzing all discharge streams containing hexane, it was possible to make an accurate hexane mass balance. It was shown that almost 80% of the total loss was due to hexane residues in the desolventized and dried meal.

INTRODUCTION

The price trend of hexane, the potential fire and explosion hazard, and environmental control regulations are factors that make it necessary to increase the knowledge about hexane recovery and hexane losses in extraction plants.

Several method for determination of residual hexane in oilseed meals and oils have been published (1-9). Attempts have been made, by these methods as well as by others, to get a true picture of the relative influence of different discharge sources. A shortcoming in published mass balances has been that only a comparatively small part of the real inventory loss of hexane has been explained (10). The difference has had to be accepted as indefinable "mechanical losses".

This report presents a reliable mass balance giving a complete and partly new picture of the importance of different discharge sources in a specific commercial pre-press-extraction plant.

The plant in which the survey was made processes ca. 800 tons of rapeseed per 24 hr. The extractor is a 24-ft Rotocel. The desolventizer-toaster (DT) is of the cylindrical, stack type. A fluidbed equipment with an air effluent of 40,000 m^3/hr is used for meal drying and cooling. A mineral oil recovery system is used for the hexane containing vent gas effluent, which amounts to 160 m^3/hr. The waste water is boiled for solvent recovery.

EXPERIMENTAL

Gas Chromatography (GC)

The GC was a dual Varian 3700 equipped with two flame ionization detectors (FID) and a Hewlett Packard 3390A integrator. One channel (A) was provided with a 1.8 m × 2.0 mm id glass column packed with 5% OV 101 on chromosorb W HP 100/120 and on-column injection. The other channel (B) had a 1.8 m × 2.0 mm id stainless steel column with the same kind of packing and a flash injector. The temperatures of the injector, the column and the detector were 160 C, 50 C and 260 C, respectively. Nitrogen was used as carrier gas at a flow rate of 30 mL/min. Attenuations employed for the GC and the integrator were 1×10^{-11} and 2^9, respectively.

Hexane in Meal

10.0 g of rapeseed meal was mixed with 20.0 mL of iso-octane and extracted for 10 min in a French "Dangoumau quantitative microgrinder", vibrating at 700 movements/min. The microgrinder consisted of a 150-mL stainless-steel container fitted with 5 steel balls of diameter 12 mm, and 20 steel balls of diameter 7 mm. The slurry was filtered and 2 μL of the solution was injected into the GC column (A).

Hexane in Oil

Two μL oil was injected into the GC column (B). The removable glass liner was provided with a glass wool plug to catch the oil.

Hexane in Water

Waste water, 100 mL, was mixed with 10.0 mL of hexane-free iso-octane and shaken for 30 min in a separating funnel. The iso-octane layer was separated and dried for 30 min over calcium chloride. Two μL of the dried solution was injected into the GC column (A).

Hexane in Air

The hexane containing gas effluent was sucked through a charcoal tube, NIOSH standard size 50/100 mg, by means of an SKC Personal Air Sampler Pump 222-3. An appropriate gas volume was chosen to adsorb 0.2-1.0 mg hexane on the charcoal. The charcoal was transferred to a test tube with screw cap, 3.0 mL of carbon disulfide was added and the mixture was shaken vigorously for 2 min. Three μL of the clear solution was injected into the GC column (A).

Standardization

Standard solutions were made from technical hexane, boiling range 65-70 C, hexane-free iso-octane (puriss) and carbon disulfide (pro analysis). New iso-octane standard was prepared every second week. The carbon disulfide standard was freshly made on the occasion of each analysis.

RESULTS AND DISCUSSION

The principal reason why correct mass balances have not been presented earlier is probably that unreliable methods for determining residual hexane in meal have been used. To find the most suitable method for this examination, extensive experiments were made, mainly by the "headspace method" (8) and a "microgrinder method" (8) which was modified in this laboratory. Table I shows a comparative study of these two methods with the very reliable but also more time-consuming "steeping method" (7) and the method by Black and Mustakas (1). The modified microgrinder method was chosen as it is the quickest, and at the same time sufficiently reliable and easy to reproduce.

TABLE I

Hexane Content in Hexane-Extracted Rapeseed Meal According to 4 Different Methods of Analysis

Method	Mean[a] hexane content, (wt%)	Time needed per analysis (hr)
Black and Mustakas (1)[b]	0.07	1.5
Steeping in iso-octane (7)	0.18	72.0
Headspace (8)	0.18	2.0
Microgrinder[c]	0.17	0.5

[a]Mean value of 14 analyses.
[b]Modified to rapeseed meal.
[c]Modification of method published by Prevot and Coustille (8).

To make the survey possible, it was also necessary to develop sampling techniques and methods for determination of hexane contents in vent gases (11) and in waste water (12).

The survey of the hexane losses was taken over a period of 142 production days. During the period, two random samples were taken each day at the meal cooler discharge. An intermittently working, automatic oil sampler was used to take one average sample each day at the oil dryer discharge. Random samples of waste water and vent air were taken once a week. The hexane contents in water and air were assumed to be constant between the sampling occasions.

During the survey period, 220 m^3 of hexane were consumed and 108,000 tons of rapeseed were extracted. Accordingly, the inventory loss was 2.04 L/ton of seed. As is shown in Table II, the total loss according to analyses was 2.00 L/ton of seed. This close agreement is probably a coincidence and the discrepancy will in the long run increase to between 5 and 10%. Even this however, means a considerable improvement compared to earlier measurements taken in this plant as well as in others (10).

A detailed investigation of the total hexane losses in the air effluents showed that they were proportional to the fluctuating hexane contents in the meal. It was also shown that almost half of the air-borne hexane was leaving the DT in a gaseous state at the meal discharge. The remaining part of the hexane discovered in the air effluents was evaporated from the meal in the meal drying and cooling steps. Consequently, the performance of the DT has to be improved not only to reduce the hexane contents in the meal but also the contents in the air effluents.

The results of the survey, and the conclusions drawn, should be applicable to any extraction plant of modern design. This means that for most plants the only way to reduce hexane losses drastically is to improve the performance of the DT.

By taking advantage of ideas put forward by Schumacher (13), concerning live steam distribution technique and desolventizing time and temperature, it was possible to improve the performance of the DT in the plant described. Thus, as a first step, the overall solvent loss was reduced from 2.0 to 1.6 L/ton of seed. To reach still better results it will be necessary to exchange the existing DT for a new one of different design.

TABLE II

Hexane Losses in an Extraction Plant According to Analyses of Different Effluent Sources

Source of hexane loss	Hexane loss (L/ton seed)	Distribution between effluent sources (% of total loss)
Rapeseed meal	1.56	78
Rapeseed oil	0.07	4
Air from dryer/cooler	0.30	15
Air from mineral oil system	0.06	3
Waste water	0.01	1
All effluents	2.00	100

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