# Development and scale up of new compressed gas extraction technologies for food and natural product processing.

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## ABSTRACT

This paper describes some of the recent research in extraction and fractionation of lipid compounds carried out at Industrial Research Limited (IRL), New Zealand, and our experience in developing a pilot scale extraction plant for demonstration of these technologies to potential end users. The processes include methods for extraction of niche high value lipids, and methods for direct processing of aqueous feed streams to produce a lipid extract and a lipid-depleted protein residue, using the food suitable and environmentally benign solvent dimethyl ether (DME).

A portable pilot scale plant was developed to encourage uptake of these processes by industry. The rationale for development of the plant was to enable potential end users to evaluate the technology without needing to invest in their own capital, to enable them to produce product samples on-site in their own registered facilities, to enable pilot studies and full scale costings to be carried out, and to showcase the technology and build familiarity and experience with the process through hands on use. Design issues that required particular attention included the need to design a versatile portable plant, and design for use with flammable solvents.

## PROCESSING TECHNOLOGIES

IRL is a Government owned research facility established with the aim of providing support to, and driving innovation for, New Zealand industry. One of the research areas is the use of supercritical fluid processing for extraction and formulation of natural bioactives for nutritional applications. This includes specialty lipids such as polyunsaturated fatty acids, carotenoids, high value seed oils, and complex lipids such as phospholipids, gangliosides, and sphingolipids. Extraction or fractionation of polar lipid, for example, can be carried out using carbon dioxide with a polar modifier such as ethanol. At around 10% ethanol concentration in $CO_2$[1] many of these lipids become soluble, but phospholipids including phosphatidylserine and phosphatidylinositol remain largely insoluble along with gangliosides. This selectivity enables effective fractionation of these components. A typical contacting arrangement is given in Figure 1.

Polar lipids can also be extracted through use of other near-critical fluids such as dimethyl ether (DME).[2-8] DME is a polar solvent with a high solvent capacity for polar lipids. Extraction can be carried out at temperatures below 60℃ and pressures as low as 20 bar. DME is non-toxic, and applications are currently in progress in Europe and New Zealand to register DME as a food grade solvent. Residual proteins are not denatured by the extraction process if the feed material is relatively dry.[2,7-8]

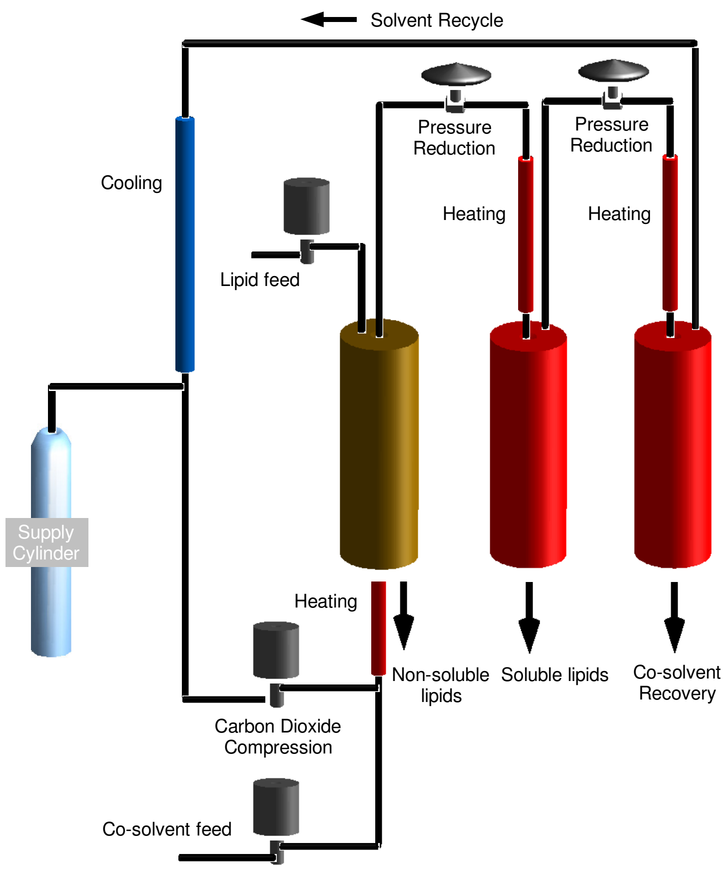


Figure 1 – Typical contacting arrangement for phospholipid fractionation using $CO_2$ and ethanol (co-solvent).

A comparison of the extraction of spray dried egg yolk powder with DME and $CO_2$ is given in Figure 2. DME extracts phospholipids from the egg yolk in addition to the triglycerides extracted using $CO_2$, and extraction is more rapid. A typical phospholipid profile for a DME extract from egg yolk is given in Table I.

DME has a vapour pressure of about 4 bar at room temperature and removal and recovery of the solvent is simple and near complete. Measurements by gas chromatography indicate residual DME levels in typical food substrates to be less than the 1 ppm detection limit after processing. DME is also mutually soluble with water and effective lipid extraction can be achieved from wet biomass without the need for pre-drying. Liquid (aqueous) feed streams can also be processed in a continuous flow contacting arrangement.

Table I – Typical phospholipids composition of egg yolk lipids extracted using DME

| **Phospholipid** | **Fraction of total phospholipids (wt%)** |
|---|---|
| Phospatidylcholine | 68.9 |
| Phosphatidylinositol | 1.8 |
| 2-lyso-phosphatidlycholine | 0.8 |
| Phosphatidylethanolamine | 23.3 |
| Sphingomyelin | 3.4 |
| 2-lyso-phosphatidylethanolamine | 1.9 |
| **Total Phospholipid (wt%)** | **13.0** |

## PORTABLE PILOT PLANT - RATIONALE

A pilot plant facility was developed to aid transfer of supercritical fluid based technologies to our industry partners. The plant was designed to be portable so that it could be installed on-site in a range of different locations and processing environments. The plant was built with the following outcomes in mind:

- **Improved industry awareness.** Installing and operating the processing equipment on site provides a tangible focus for management to relate to when engaging in further development research, and gives operators a hands on feel for using the technology.
- **Minimisation of industry investment in fixed capital.** The cost of investing in one-off pilot scale equipment for process development is often a barrier to industry uptake of new technologies. Development of a pilot facility that can be leased to a number of companies removes this investment barrier.
- **Concept demonstration.** The plant is a showcase for supercritical fluid processing and can be used to demonstrate the feasibility and effectiveness of the process directly to industry clients.
- **Scale up and costing data.** The pilot plant is intended to provide data at an intermediate scale suitable for use in development of costing models for a full or larger scale process.
- **Process development.** The plant is expected to be flexible enough to allow a wide range of processing options to be investigated under a wide range of operating conditions, enabling rapid development of optimised processing opportunities.
- **Production of food grade product samples.** Operation of the plant inside registered food production facilities enables food grade samples to be produced that can be used directly in consumer or efficacy trials. This allows development work to be carried out without the research laboratory itself needing to establish and maintain registered facilities.

The pilot plant was completed in 2007 and has exceeded these expectations to date. The plant has been used by a client company in the development of a new food product that required production of several hundred kilograms of food grade trial product for use in efficacy trials. Use of the portable pilot plant enabled this to be carried out. The processing technology now has a much higher profile within the client company enabling further developmental work to be carried out.

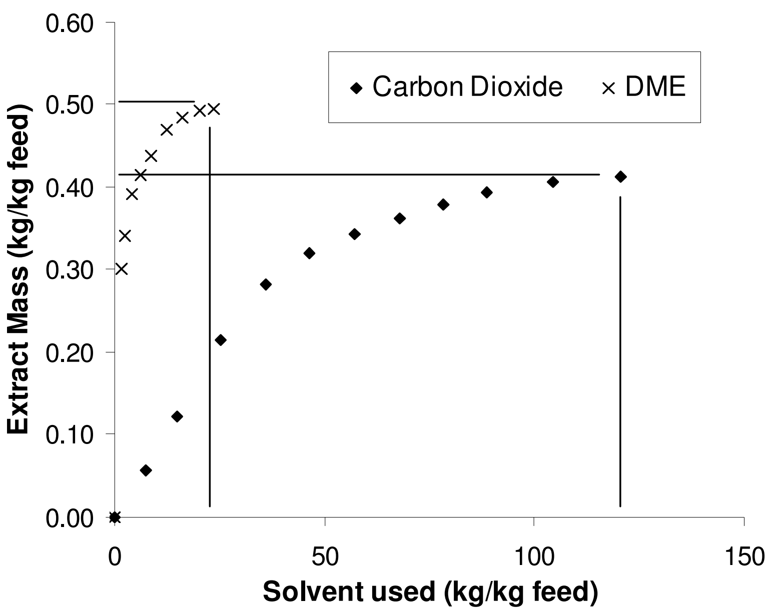


Figure 2 – Extraction curves for spray dried egg yolk powder using DME and $CO_2$.

The pilot plant has also been used in development of a new process for a product in the building sector. Use of the plant has enabled laboratory scale experiments to be transferred to the pilot scale and a realistic commercial process to be developed with minimal capital investment and within a period of 2-3 months. This was facilitated by the flexibility of the plant, enabling a range of processing options to be assessed and optimised.

Development of the pilot plant had unanticipated benefits, including attracting favourable pricing from some of the equipment suppliers on the basis that the pilot plant would be a showcase for the equipment and a prototype for potential larger scale installations. The portable pilot plant has also enabled additional work at IRL to be carried out when the plant is not otherwise in use.

**PORTABLE PILOT PLANT - DESIGN**

The plant was built to the following specifications:

- Two 10 litre extraction vessels to enable processing of up to several hundred kg of product in a reasonable time frame.
- Two stage separation.
- Operation up to 350 bar pressure.
- Solvent flow up to 25kg/hr
- Co-solvent flow rate up to 2L/hr
- Liquid feed or additional co-solvent flow up to 10L/hr
- Capability for upflow, downflow, continuous liquid feed processing, and continuous processing of powder feed materials and solid extracts by alternating between vessels.
- Rated for use with flammable solvents, such as ethanol and dimethyl ether.
- Self contained. Able to fit inside a standard shipping container.
- Fully customizable control but able to be fixed for specific applications.

Figure 3 – Portable pilot plant in use on an industrial site.

The plant (Figure 3) requires basic services to be supplied by the user – air, chilled water, steam, and electricity - but is otherwise self-contained. The overall footprint of the plant is 3 m by 1.2 m. It stands 4 m high when in use but packs to less than 2.1 m.

All of the pumps and valves on the plant are air driven, and the instrumentation is designed for use in a Zone 1 (IEC 60079 standard) hazardous environment. The plant can generally be operated on less than 100kg of solvent, which falls below the level for which mandatory handling procedures are required for use of flammable solvents in New Zealand. In practice however, the plant is still required to meet the standard of the best codes of practice. Use of the plant on a remote site also places some operational requirements on the industry client for location and use of the plant, and for handling of the flammable materials.

The design of the plant required a tradeoff between operational flexibility and simplicity for cleaning and sterilization. Flexibility of operation was the primary requirement in this case, but the plant was designed to meet typical food guidelines for minimization of dead volumes, and use of smooth finishes and food suitable materials.

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