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OPTIMIZATION OF PROCESS WATERS FROM A FIELD PEA FRACTIONATION PLANT

by


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Dedicated to my loving Grandparents,
Mrs. Berta Stengert and the late Mr. Reinhold Stengert.

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First, I would like to thank Dr. R.A. Gallop for for his encouragement, patience and knowledge during my Bachelor's and Master's studies. He has instilled in me greater insight and appreciation for human potentials, and a deep concern for the fragile little planet we call home.

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ABSTRACT

Eight sources of process effluents from a field pea fractionation facility in Portage la Prairie, Manitoba were characterized. The centrate waters from the primary desludgers were found to have the highest overall organic loading and total nitrogen content. The effluent from this process was chosen for subsequent component and water recovery trials using lab scale ultrafiltration (UF) membranes and pilot scale reverse osmosis (RO) membranes.

Ultrafiltration of the centrate waters using 10,000, 30,000 and 50,000 molecular weight cut-off (MWC) polysulfone membranes was shown to be an ineffective method of recovering components, as over 50% of the organic solutes and nearly 100% of the inorganic solutes were passed with the permeate. Using the 10,000 and 30,000 MWC membranes, retention of protein was greater than 90%. However, less than 10% of the total solids in the concentrate fraction was found to be true protein.

Severe fouling of the hollow fibers occurred using the 30,000 and 50,000 MWC membranes, but was less evident in the 10,000 MWC trials. A pretreatment of the feedwater using powdered activated carbon did not effectively reduce the flux loss encountered during ultrafiltration.

Reverse osmosis treatment of the desludger centrate using 89%, 92% and 97% rejection membranes resulted in a

four fold concentration of the effluent solutes as a concentrate fraction. The total solids in this fraction was comprised of approximately 75% carbohydrate, 15% ash and 3% protein. The permeate fracions possessed an average organic content of 125 mg/L and an inorganic content of 900 mg/L. All of the permeate fractions were relatively free of colour and turbidity. Additional trials using effluent water from the fiber isolation process, as the RO feedwater source, yielded proportionately similar results.

Extensive membrane fouling caused reductions in permeate flux of more than 50% during all RO trials. Several manufacturer recommended cleaning solutions were inadequate in restoring the membrane flux lost from fouling.

Laboratory scale studies showed that the reuse of primary desludger effluents in the protein isolation process reduced protein recovery by less than 3%. Reuse of effluent from the secondary protein desludgers resulted in a 2% increase in protein recovery. Reuse of these and other plant streams would decrease water usage as well as conserve energy and chemical resources used to condition these waters for plant operations.

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1. INTRODUCTION

Woodstone Foods Limited is a pea fractionation facility located in Portage la Prairie, Manitoba. This plant utilizes a wet process to mill and fractionate prairie grown yellow field peas (Pisum sativum var. century) into a variety of food ingredients. Although Woodstone has created ingredient blends with raw materials such as corn, wheat and skim milk powder, pea products account for the vast majority of company sales. Using technology developed at the government-run POS Pilot Plant in Saskatoon, Saskatchewan, Woodstone was set up to capitalize on the vegetable protein market boom that occurred in the 1970's.

In addition to protein isolates, fractionation of this pulse crop yields starch and two types of fiber concentrates. The starch fraction produces a gel that is stronger and more opaque than conventional sources. Fiber concentrates are derived from the seed coat and the intracellular walls of the cotyledon. Due to the unique physical and chemical properties of these products, they have found a market in the baking, meat, condiment and pharmaceutical industries.

Unfortunately, while the wet fractionation method produces component concentrates with purities superior to that of air classification, large quantities of water are used in, and discharged from the processes. Processes contributing the largest volumes of water and the highest

concentration of environmental contaminants are usually targeted for treatment.

Listed below are Woodstone's major plant effluent sources and their respective approximate outputs.

1) **Pea wash.** At this station, water is used to clean foreign matter from the split peas before grinding. Larger particles of pea hull, pod, dirt and insects carried by this water are removed by a screen before discharge. The mean output of this water is 18 L/min.

2) **Primary protein desludgers.** This pair of industrial size centrifuges remove precipitated protein from the process water. High concentrations of solutes remain in solution after protein removal. The discharge rate of the supernatant is approximately 60 L/min for each desludger.

3) **Secondary protein desludgers.** The protein sludge from the primary desludgers is mixed in tanks with fresh water to wash the protein. The secondary centrifuges remove the washed protein from the suspension. Supernatant is discharged at a rate of 72-85 L/min per unit.

4) **Starch decanter.** This device decants, or pours off the water carrier leaving the heavier starch granules to be pumped from the bottom for further processing. The decanted water is discharged from this process at 81 L/min.

5) **Cellular fiber decanter.** Similar in function to the starch decanter, the fiber decanter also discharges a mean of 81 L/min.

6) **Outside effluent tank.** Originally intended for fermentation treatment of the effluent, this tank acts to mix all plant waters before discharge to municipal sewers. Samples drawn from this site are considered to be composite samples. The discharge equals the sum of all waters released from plant activities (>500 L/min).

As the paired primary and secondary protein desludgers possessed different operating efficiencies, they were reported as individual effluent sources.

With increasing costs for water purchase and disposal, a growing awareness of environmental concerns and pressure to reduce discharges, Woodstone Foods has initiated work in the water treatment/product recovery areas. Previous efforts included the purchase and attempted use of industrial scale ultrafiltration (UF) and reverse osmosis (RO) systems. These units held spiral wound polysulphone and cellulose acetate membranes, respectively. Use of this equipment was not successfully accomplished as an immediate and almost complete fouling of the membranes in both units occurred when subjected to plant process effluents.

The studies presented in this thesis attempt to provide information on an array of Woodstone's water treatment concerns:

i) Composition of process effluent streams

ii) Application of UF to effluent streams
   - necessity of UF prior to RO?
   - optimum molecular weight cut-off (MWC)
   - composition of UF permeate and concentrate

iii) Application of RO to effluent streams
   - membrane flux performance
   - composition of RO permeate and concentrate

iv) Protein recovery from effluents via UF/RO

v) Composition and prevention of membrane fouling

vi) Feasibility of treated effluent recycle

Although many contraints make it difficult to provide conclusive answers on all these concerns, the research

performed was intended to provide insight and direction to future research and process modifications.

2. REVIEW OF LITERATURE

2.1. Field Pea Production and Utilization

2.1.1. Provincial Field Pea Production

Field peas (Pisum sativum L.) are currently being evaluated as a high protein crop for the Canadian prairies. This is due to an increased interest in plant proteins as components in agricultural and industrial products (Sumner et al., 1981).

Approximately 58,700 hectares of Manitoba farmland was seeded with smooth yellow field peas in 1986 (Manitoba Department of Agriculture, 1986). This value was up 45% from 1985 and represents 45.0% of the field peas grown in Canada. Although several varieties of field peas are grown in Manitoba, the majority of the 103,400 tonnes harvested were of the Century and Trapper varieties.

2.1.2. Field Pea Processing

An economic trend has shifted users of the traditional non-fat dry milk protein to an array of less expensive vegetable protein sources (Delaquis, 1983). A novel alternative, pea protein, may partially displace the more common vegetable sources such as soy, cotton seed, fababean, peanut and canola in food and feed products. In addition, a larger secondary fraction, high in starch, is obtained during pea protein isolation (Sumner et al., 1981). The

economic feasibility of pea protein isolation would depend, in part, on the acceptability of this starch fraction in the replacement or supplementation of more established starch sources such as corn, potato and rice.

Field peas are especially suited for use in protein and starch concentrate production due to their high lysine content and relatively low cost (Sosulski, 1982). The low concentrations of anti-nutritive compounds presents no problems in the consumption of raw or cooked peas. Also, dried field peas have a loose hull that can be easily removed and constitutes only 8% of the seed mass (Reichard and MacKenzie, 1982).

Present day commercial use of pea protein isolates include that of filler, extender and nutritional supplement in products such as sausages, soups, sauces, breads, beverages, non-dairy frozen deserts, health food items and animal feeds.

2.1.3. Field Pea Composition

McWatters and Cherry (1977) reported the total carbohydrate content of dehulled field peas to be 59.7%. The majority (46.6%) of carbohydrate present is starch, with an amylose content of approximately 33% (Biliaderis et al., 1980). The remaining portion primarily contains the polysaccharides hemicellulose, cellulose, lignin, gums, pectins, mucilages and -galactosides verbascose, stachyose

and raffinose. According to Bhatty and Christison (1984) total sugars represent 8.04% of the pea cotyledon consisting mainly of oligosaccharides (5.88%), sucrose (2.04%) and glucose (0.12%).

Sumner et al. (1981) obtained a total lipid concentration of 2.1% in dehulled peas. It was also reported that most of the endogenous fat associated with the pea endosperm was absorbed by the pea protein isolate during wet processing.

Many researchers reported similar ash concentrations between 2.4 to 3.4% in dehulled peas (Sumner et al., 1981; McWatters and Cherry 1977; Colonna et al., 1980; Bramsnaes and Olsen, 1979; Sumner et al., 1979; Sosulski, 1979; Bhatty and Christison, 1984; Reichert and MacKenzie, 1982). According to Reichert and MacKenzie (1982) the four major elements present in the ash were potassium (1.04%), phosphorous (0.39%) magnesium (0.10%) and calcium (0.08%).

2.1.4. Pea Fractionation Methods

Presently, there are two commercial processes being used to fractionate field peas. The most common method is air classification. This method employs a current of air to separate pea protein from the starch components based on differing particle density and mass. Reichert (1982) used air classification to fractionate dehulled Trapper field peas ranging from 14.5 to 22.5% protein content. The

resulting protein concentrates contained 33.6-60.2% protein. Thus, this method yields protein fractions of relatively low purity. A commercial facility for the air classification of field peas has been established at Saskatoon, Saskatchewan.

A second means of producing pea protein fractions is a wet process using large quantities of food grade water. In this method, solubilized pea proteins are precipitated out of an aqueous solution by isoelectric pH adjustment. The protein isolate is removed from the suspension by centrifugation. The resulting protein sludge is washed with fresh water and re-centrifuged prior to spray drying. The fiber and starch fractions obtained prior to protein precipitation are separated before decantation of associated carrier/wash waters. Drying of the fiber and starch fractions can be accomplished by spray or drum drying. All plant process waters are combined prior to discharge into the municipal sewer system. Woodstone Foods located in Portage la Prairie, Manitoba currently fractionates field peas using a wet process.

Other vegetable protein extraction methods exist but have not been used for the commercial preparation of field pea protein concentrates. Methods of interest include:

1) salting out of solubilized proteins by ionic strength adjustment (Murray et al., 1978),
2) extraction of protein using heat denaturation (Ohren, 1981, and Pepper and Orchard, 1981), and

3) ultrafiltration of protein extracts (Lawhon et al., 1977, and Nichols and Cheryan, 1981a,b).

The former and the latter extraction techniques likely have the greatest potential for the production of vegetable proteins with high purity and superior functional characteristics.

2.2. Ultrafiltration and Reverse Osmosis

2.2.1. History of Membrane Development

Commercial utilization of reverse osmosis (RO) and ultrafiltration (UF) processes have been known for more than 100 years (Lacey, 1972). Only in the last 20 years has membrane technology advanced to the point of economic utilization of the relatively new science (Applegate, 1984). Application of these filtration processes to the food industry for the purification, separation and concentration of food components is increasing rapidly.

The development of ultrafiltration membranes was consequential to the advent of RO membranes. Smaller pore size was the only structural difference distinguishing early RO membranes from UF membranes. Hence, the classification of any membrane into an RO or UF category was mainly undertaken to describe the general separation capability of that membrane (Sourirajan and Matsuura, 1985). Recent variations between RO and UF membrane composition, configuration and construction is a result of suiting

application requirements rather than satisfying the basic mechanism of operation.

The present water desalting process utilizing semi-permeable membranes was conceived in 1956 (Sourirajan and Matsuura, 1985). Reid and Breton (1959) reported the development of a cellulose acetate membrane at the University of Florida. However, due to the high density of these initial membranes, they were labelled impractical for commercial desalination processes. Loeb and Sourirajan (1960) developed the cellulose acetate membrane with assymetric densities possessing high permeate production rates required for commercial exploitation (Sourirajan and Matsuura, 1985). Based on a preferential sorption capillary flow mechanism, this process was later named reverse osmosis.

In 1970, synthetic membranes derived from an aromatic polyacrylamide polymer (aramid) were commercialized by Dupont (Applegate, 1984). These new membranes were less susceptible to chemical and biological attack than the organic cellulose acetate type. However, the polyacrylamide membranes were found to be more susceptible to chlorine degradation than the organic membranes.

Cabasso et al. (1979) were among the first researchers to work on the development of the polysulfone membranes. Although aliphatic and aromatic polysulfones have been synthesized, only the aromatic compounds possess a molecular

weight high enough to be suitable for incorporation into RO or UF membranes. This membrane material has proven to be very resistant to biological, chemical (including chlorination) and thermal degradation and has great potential for future membrane applications.

A thin film composite membrane based on a fine layer of polymeric amine, supported by a polysulfone substructure, was introduced in 1977 (Applegate, 1984). These membranes possessed similar characteristics to previous aramid types, but were even more susceptible to chlorine degradation while producing higher permeate flow rates.

More recently, inorganic membranes have been developed from materials such as ceramic, carbon and sintered metals (Thomas et al. 1986). These new membranes allow great flexibility in the type of products processed and methods of cleaning and sterilization. The literature available on applications of inorganic membranes is still very limited, but is expected to grow rapidly.

2.2.2. Food Plant Membrane Applications

The majority of membranes in use today are employed in the dairy industry and for potable water production from salty and brackish water. However, new applications are increasing in number and include fruit juice concentration, sugar concentration, protein and enzyme recovery as well as by-product recovery and wastewater treatment.

Ultrafiltration and/or reverse osmosis can be utilized in many wet processes for the purification, separation or concentration of food components, food process waters, or potable water (Parkinson, 1983). Whether UF, RO or both processes are used is dependent on the objective of the processing and nature of the fluid being used. These two factors also influence the choice of membrane type, membrane configuration, system size and pretreatment requirements.

Larson (1984) predicted that by 1990 an estimated 650 million L/day installed RO membrane capacity would increase to more than 2000 million L/day. Commercial membrane systems can range from 400 L/day capacity units used by laboratories to 100 million L/day plants used for municipal water-distribution systems (Applegate, 1984).

Although, the literature available on membrane processing of fluid foods and waters, other than dairy products, is somewhat limited, the advantages of this technology are well recognized. Cicuttini et al. (1983) described an RO process used to lower the energy consumption at a corn milling plant. Gooding (1985) estimated that membrane concentration requires only 1-10% of the energy used in conventional thermal evaporation processes.

Ultrafiltration has been used to separate desirable fractions from non-desirable ones. The non-desirable fractions may consist of food components that cause sensory or functional defects in a food product or play anti-

nutritional roles on consumption. Swientek (1984) described how ultrafiltration is being used to remove lactose and water from milk in the production of mozzarella and other semi-hard and soft cheeses. Omosaiye et al. (1978) reported that the levels of oligosaccharides in soybean waters could be considerably reduced using UF. Oligosaccharides are known to cause gastrointestinal problems (flatulence) when consumed by humans and non-ruminant mammals.

UF can also be used to recover an extract of high molecular weight components such as starch, protein and enzymes. Pepper and Orchard (1981) described a RO system for the treatment of potato starch effluent. Chiang et al. (1986) showed that UF and RO could be used to recover mushroom components from process waters for use as food or feed products.

To date, no reference to the use of RO or UF membranes in the processing of effluent from field pea fractionation could be found in the literature. It is difficult to extrapolate the available technological information to such effluent because this pulse crop is unique in its compositional qualities. Small variations in the chemical or physical characteristics of a membrane substrate may induce considerable changes in a system's effectiveness (Sourirajan and Matsuura, 1985). Therefore, information on the compositional character of all fluids to be processed is required prior to the evaluation of a membrane system.

2.2.3. Membrane Treatment of Process Waters

Ultrafiltration and reverse osmosis systems have applications in water treatment processes due to their powerful retention properties, speed of processing and relatively low energy consumption. Membrane treatment of process waters may serve one or more of the following functions:

1) Recovery of water for in plant reuse
2) Purify water to reduce sewage surcharges and/or environmental burdens of discharge
3) Recover valuable by-products
4) Pretreat the water for additional processes.

Certain chemical analyses of process waters can aid in evaluating the need for membrane treatment. Biological oxygen demand (BOD) and chemical oxygen demand (COD) are two parameters commonly used to measure the concentration of organic matter in a wastewater stream. The level of organic constituents present in a water will dictate the degree of treatment required for biodegradation by a municipal treatment facility or when discharged into the environment. The level of suspended solids is often used to determine sewage treatment surcharges. Penalties are normally levied for BOD and suspended solids concentrations higher than 300 and 350 ppm respectively (City of Winnipeg, 1973).

The literature contains several references to the membrane treatment of effluent waters from sources other than field pea processing. Spatz (1973) reported that a 99% reduction in BOD, present in candy process water, could

be achieved using spiral-wound cellulose acetate RO membranes. Similar results were recorded for a maraschino cherry production line where both sugar and dye were removed for reuse (Spatz, 1975). The RO permeate was found to be suitable for recycling in plant processes.

Lawhon et al. (1973, 1977) used flat sheet and spiral wound UF and RO membranes to obtain a protein concentrate and purified water fraction from cottonseed whey. Cicuttini et al. (1983) also used a tubular system to process water from a corn wet milling operation. Both papers suggest that the high quality permeate water resulting from membrane treatment can be reused in plant processes.

Pepper and Orchard's (1981) work on potato starch isolation effluents demonstrated that a 94% reduction in plant water consumption could be realized using RO permeate recycle. The permeate possessed a residual COD of 400 mg/L. The resulting potato protein concentrate was recovered as a valuable by-product. Chiang and Pan (1986) showed that an RO system could reduce BOD and COD in potato process waters by 99 and 98% respectively. In addition to permeate recycle benefits, the RO concentrate fraction was found to have a high protein, sugar and mineral content making it suitable for use as animal feed.

Wu (1986) found that spiral wound UF and RO membranes could be successfully used to separate wheat-stillage solubles into a large volume of permeate water for reuse or

safe discharge and a small volume of concentrate with food grade by-product potential. Several researchers have also demonstrated the advantages of membrane systems in the processing of soy extracts and process effluents. Bramsnaes and Olsen (1979) recommended the use of air classification for the production of soy protein fractions, as wet fractionation operations produce large volumes of heavily organic laden wastewaters. Lawhon et al. (1977) found that soy extracts, when processed with UF and RO, would be lower in total solids than the native surface waters. However, results presented by Nakao et al. (1983b), using effluents from a miso (fermented soy) factory, showed BOD reductions of only 12-62% using UF and 83% with RO. It was concluded that the flat sheet cellulose acetate membrane had a low rejection of soy fermentation products such as organic alcohols, acids, ketones and aldehydes.

2.2.4. Membrane Fouling

One parameter dictating the efficiency and consequent feasibility of a commercial RO or UF system is membrane flux. The most desirable membranes would possess high permeate production rates and a low susceptibility to fouling. Cellulose acetate membranes have been shown to lose 5-10% of their initial flux due to irreversible fouling and membrane compaction (Osmonics, 1984). An additional loss of 20% can be expected over the life of the membrane. The fouling of membranes by organic or inorganic

constituents present in commercial food plant waters can produce many undesirable effects:

1) Increased process time
2) Decreased permeate/concentrate production
3) Higher energy consumption
4) Increased solute rejection
5) Higher chemical cleaning costs
6) Increased labor
7) Shortened membrane life
8) Increased equipment wear
9) Costly system scale up

Richter (1983) reported that fouling of membranes by proteins and salts is the most significant problem in dairy ultrafiltration. Garontte and Amundson (1982) found that the permeate flux obtained during the UF of whole milk declined to 0 L/h before a 5X concentration factor was reached. It was suggested that this was due to formation of a gel or precipitate layer of macromolecules on the membrane surface of the hollow fibers.

Work by Chiang and Pan (1986) has shown fouling of flat sheet RO membranes by pectin-like substances found in waters from a sweet potato starch process. Reverse osmosis of these waters was reportedly feasible when preceeded by ultrafiltration using hollow fiber membranes. Similar results were obtained by Chiang et al. (1986) when mushroom blanch water was membrane processed with UF and RO. However, no suggestion as to the type of component causing the fouling was made.

Several researchers have worked on membrane processing of soy extracts. Lawhon et al. (1977) observed flux losses

of greater than 75% when soy slurries and UF extracts were processed using a RO system. Ultrafiltering the soy extract slurry did not prevent a considerable decline in flux due to fouling. Omosaiye et al. (1978) reported a similar loss in flux due to fouling of the hollow fibers in the UF system used. Little overall difference in the rate of fouling was noted when the extract was processed in the pH range from 2 to 10. In another paper, Omosaiyie and Cheryan (1979) suggested that discrepancies in the mass balance of UF fractions could be attributed to the deposition of components in the lumen of the UF hollow fibers. This occurred despite prefiltration of process fluids with a plate and frame filter press. Nichols and Cheryan (1981a,b) attempted to concentrate proteins from a prepared soy extract using polysulfone and acrylic vinyl copolymer hollow fiber membranes. Some severe fouling was encountered. The copolymer membranes showed considerably more protein adsorption than the polysulphone. These researchers suggested that a soy extract must be low in fiber and other insoluble carbohydrates in order to use UF for protein purification and concentration. Failing this would result to a certain extent, in poor UF unit performance. Yamauchi (1982) stated that experimental results of soybean and egg albumen ultrafiltration did not agree with those predicted by a concentration polarization gel model. It was postulated that a hard gel model would explain interactions among protein molecules in the gel layer which might cause much lower than predicted permeate flux. Nakao et al. (1983a,b)

used tubular cellulose acetate UF membranes to process cooking waters from a miso (fermented soy paste) factory. Permeate flux was found to decrease rapidly (within one hour during UF and three hours during RO) due to the formation of a gel layer on the membrane surfaces. This layer also acted to reject solutes in both UF and RO systems. It was composed of rigid chain polymers of high molecular weight polysaccharides and spherical globulin proteins. Nichols and Cheryan (1981b) suggested that it was difficult to compare data regarding membrane flux since most results were not reported in terms of a universal parameter such as membrane permeability coefficient. Instead each membrane system and feed water must be evaluated on an individual basis.

2.2.5. Membrane Cleaning

The literature is notably deficient in references pertaining to the cleaning procedures and materials required for the proper cleaning of fouled membranes.

Cleaning is required to remove organic and/or inorganic materials present in the concentration polarization and fouling layers on the surface of membranes. Additional deposits of these materials may be found plugging membrane pores and in the matrix of the surface and support structure (Sourirajan and Matsuura, 1985). Ineffective removal of the deposits can lead to acute or gradual loss of

permeate flux. In addition, up to 30% of permeate flux losses may be expected over the life of a membrane due to membrane compaction and irreversible fouling (Osmonics, 1984).

Cleaning is necessary when excessive deposits occur due to the accumulation of suspended and colloidal constituents, precipitation of soluble salts, or biological growth occurs (Dudley, 1971). Thus, it is beneficial to first identify the major contributing factors to the fouling phenomenon.

As mentioned in section 2.2.2. membrane performance is difficult to predict when the feedwater solutions consist of complex mixtures of food constituents rather than an ideal model solution. Similarly, the ability of a particular cleaner or cleaning procedure to restore lost permeate flux is rather unpredictable due to the specific nature of the fouling layer. Membrane cleaning programs must be developed to suit each particular application to ensure effectiveness of cleaning and compatibility with membranes (Anon, 1984). The use of compatible cleaners and conditions is necessary to prevent degradation of the membrane resulting in solute rejection and flux losses. Frequency of cleaning is dependent on the fouling rate and may vary from once every few hours to once a day for food products (Hedrick, 1983). Factors affecting the cleaning cycles according to Hedrick:

1) Membrane composition, durability and configuration
2) Nature and amount of deposits on the membrane
3) Quality of the cleaning water (e.g. hardness)

4) Type of detergents (e.g. enzymatic, acid, alkali)
5) pH of cleaning solution
6) Temperature of solution
7) Purity and temperature of rinse water
8) Contact time and velocity of cleaning/rinsing solution
9) Government regulations

Cleaning programs are considered successful if the permeate flux is completely restored after each cleaning cycle and negligible loss in rejection properties occurs from the program. As the effective cleaning of membranes is considered a limiting factor in food applications (Harper and Moody, 1981), a program must be developed prior to commercialization of the process.

3. MATERIALS AND METHODS

3.1. Effluent Characterization

3.1.1. Sample Sources

Seven plant effluent streams were selected for a characterization study. These included the waters discharged from the pea wash station, primary and secondary protein desludgers and the starch and fiber decanters. An eighth source (outside effluent equalization tank) was available as a composite of all plant process waters discharged. As the paired primary and secondary protein desludgers possessed different operating efficiencies, they were characterized and reported as individual effluent sources.

3.1.2. Random Sampling Study

3.1.2.1. Sampling Method

In this study, samples from the pea fractionation effluent sources were collected for analysis. Four sets of samples were obtained on random dates between September, 1983 and February, 1984. Samples, 4 L in size, were drawn from the indicated sources at Woodstone Foods in Portage la Prairie and transported on ice to the Food Science Department, University of Manitoba. On arrival the samples were stored at refrigerated temperatures ($4^{o}C$) until subsequent analysis, within two days.

3.1.3. Multiple Sampling Study

3.1.3.1. Sampling Method

Effluents from the protein desludgers were chosen as the test feedwaters for subsequent membrane studies due to their high total solids content and rate of discharge. An extended sampling plan was devised to provide an indication of compositional variation of these effluent streams. Four liter samples were drawn hourly, for six hours, from each of the four protein desludgers. A 200 liter barrel was filled with effluent to provide a representative pool for sample withdrawal. The pH and temperature of the samples were checked immediately, followed by transport to the Food Science Department for analysis.

3.1.3.2. Sample Analysis

In the multiple sampling experiment, two sets of effluent samples drawn from Woodstone Foods were subjected to the following analyses:

1) Organic loading (COD)
2) Carbohydrate
3) Tannin
4) Total nitrogen
5) TCA precipitable nitrogen
6) Total solids
7) pH
8) Temperature

3.2. Chemical Analyses

Chemical analyses were performed according to methods found in Standard Methods for the Examination of Water and Wastewater, 16th edition, 1985. APHA, AWWA, WPCF.

Analysis	Method Number
Ash	209D
Chemical Oxygen Demand	508C
Chloride	407A
pH	423
Semi-Micro Kjeldahl Nitrogen[1]	420B
Suspended Solids	209C
Tannin	513
Total Solids	209A

[1] 15th ed. 1980

Carbohydrate was initially determined by the Dubois et al. phenol-sulfuric acid test as outlined by Benefield and Randall in Water and Sewage Works, February, 1976. Carbohydrate was later calculated by difference (ie. total solids - protein - ash = carbohydrate) when ash analysis was performed.

Protein was determined by precipitation with 10% trichloroacetic acid (TCA), followed by Kjeldahl analyses on the resulting protein pellets obtained on centrifugation. To aid in the quantitative transfer of the protein, the pellet was solubilized in 0.1N NaOH prior to Kjeldahl digestion.

3.3. Ultrafiltration Trials

3.3.1. Equipment

The laboratory scale UF unit used is this study was a Model DC-2 hollow fiber concentration/desalting system supplied by the Amicon Corporation of Lexington, Massachussetts. This unit includes a 2 L feedwater chamber, dual head peristaltic pump, prefilter, variable back pressure valve, flowmeter, and pressure safety device. It is recommended for solutions with solute concentrations in the range of 0.4 to 20%.

The DC-2 system houses one 20.3 cm long hollow fiber module containing 55 fibers with an internal diameter of 1.1 mm. Each module contains 0.03 m^2 of membrane area. The 10000, 30000 and 50000 MWC modules used in these experiments were supplied by the Amicon Corporation.

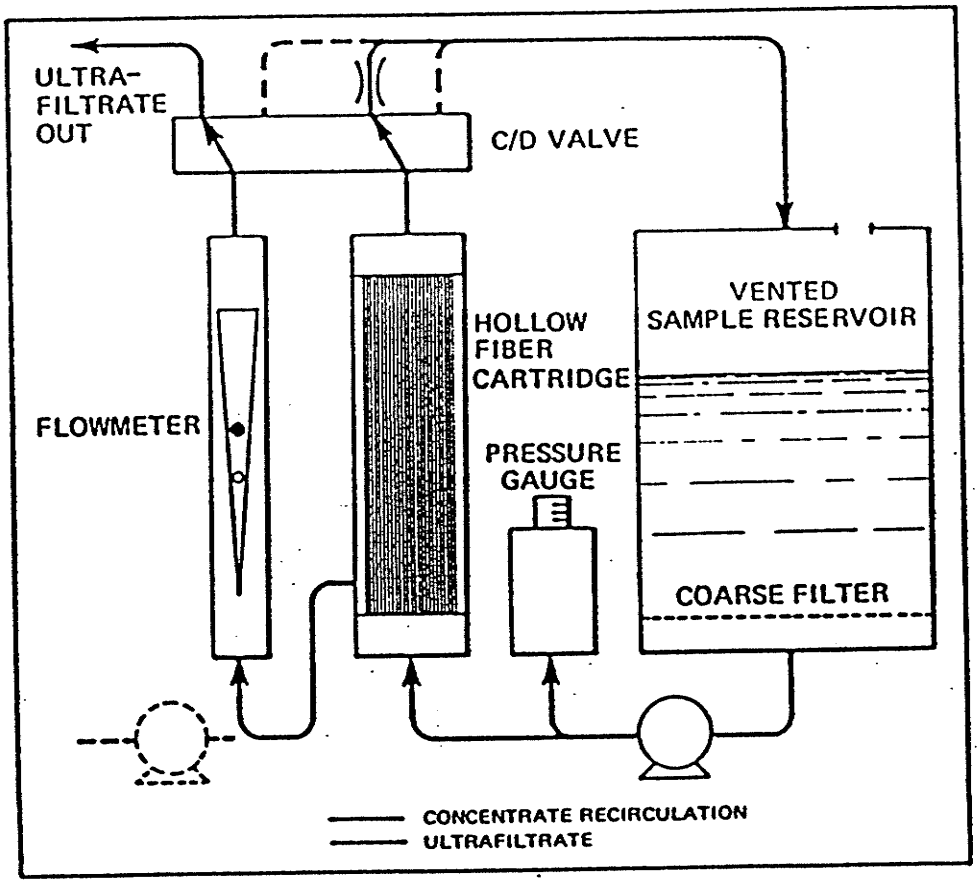
A temperature controlled water bath was used to maintain a constant feedwater temperature at 45^{o}C.

3.3.2. Operating Conditions and Methods

A total of 14 ultrafiltration trials were undertaken. The primary protein desludger effluent was used as the feedwater source in these trials. Effluent samples were collected from the desludgers and tested for pH and temperature prior to use.

Figure 3.1. illustrates the scheme used in all UF

Figure 3.1. Schematic of Amicon Model DC-2 ultrafiltration unit.


ULTRA-
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VENTED
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FIBER
CARTRIDGE
FLOWMETER
PRESSURE
GAUGE
COARSE FILTER
CONCENTRATE RECIRCULATION
ULTRAFILTRATE

trials. Selected samples were poured into a two liter Erlenmeyer feedwater flask and placed in a temperature controlled waterbath. The bath was regulated at 40°C to approximate plant process conditions. Plastic tubing was used to connect the feedwater flask to the Amicon UF unit pump. A return tube was connected from the units concentrate port to the feedwater flask. When operational, the Amicon unit pumped feedwater from the Erlenmeyer flask through the prefilter to the hollow fiber module. Permeate that has passed through the fiber wall leaves a separate port to be collected in a one liter beaker. The concentrated feedwater that exited the lumen of the fibers was pumped back to the feedwater flask via the return tube. Unit operation was continued until the desired volume concentration factor had been reached. One or two liters of the effluent feedwater was concentrated to approximately 100 mL in the 10X and 20X concentration trials respectively. The operating pressure was regulated at 10 psi using the variable backpressure valve.

3.3.3. Feedwater Pretreatment

Three types of feedwater pretreatment were used. Coarse filtration using the pre-filter mounted on the Amicon unit was the first pretreatment evaluated. No retention specifications were supplied with the filter supplied by the manufacturer. This simple pre-treatment was used on UF trials with 10,000, 30,000 and 50,000 MWC hollow fiber

modules.

A second type of pre-treatment consisted of filtering the feed effluent with Whatman #5 paper prior to processing. This paper has a 6.5 micron particle retention rating for the removal of suspended solids. Filtration of the feedwater was accomplished with the aid of an 11 cm Buchner funnel, a two liter vacuum flask and an aspirator. The trials were performed using the 30,000 and 50,000 MWC modules.

The third pretreatment involved mixing two liters of fresh effluent with two grams of powdered activated carbon. After two minutes the suspension was vacuum filtered through Whatman #5 filter paper. The filtrate was used as UF feedwater for trials with the 30,000 and 50,000 MWC modules.

3.3.4. Sample Analysis

The ultrafiltration feedwater and fraction samples were analyzed for the following parameters:

i) COD
ii) Total solids
iii) Carbohydrate content
iv) Total nitrogen
v) Protein
vi) Tannin
vii) Ash content

3.3.5. Flow Rate Measurement

The permeate flow rate was determined by collecting permeate from the UF unit in a graduated cylinder for

exactly five minutes. The volume of permeate collected, multiplied by 12, and divided by the membrane area represents flux in $L/m^2/hr$. A flow rate measurement was performed every 30 minutes. All measurements were taken at an operating temperature of 45^oC.

3.4. Reverse Osmosis Studies

3.4.1. Equipment

The reverse osmosis unit used in these studies is shown in Figure 3.2. The pilot scale RO system, model 1000 GPM, was supplied by Ajax International Corporation of Santa Barbara, California. This system includes a high pressure centrifugal pump, prefilters, a pressure vessel, pressure gauges as well as pressure and recycle control valves. The pressure vessel houses up to two spiral wound membrane modules each 9.5 cm in diameter and 96.5 cm in length. Modifications of the vessel were required in order to accommodate the 5 cm diameter modules used in these experiments. Table 3.1. lists the three types of spiral wound modules used in these studies.

A plate heat exchanger, Model P5 VRB was used to cool down the desludger effluents prior to RO. The manufacturer of this piece of equipment is unknown. Cold plant water was used as the heat exchange media.

A line pressure of 2.4 kg/cm^2 was supplied to the Ajax

Figure 3.2. Reverse osmosis system diagram.

1. Feed Water Tank
2. Feed Pump
3. 5 micron Prefilter Cartridges
4. High Pressure Pump
5. R.O. Module
6. Permeate Tank
7. Back Pressure Valve
8. Concentrate Valve
9. Concentrate Tank

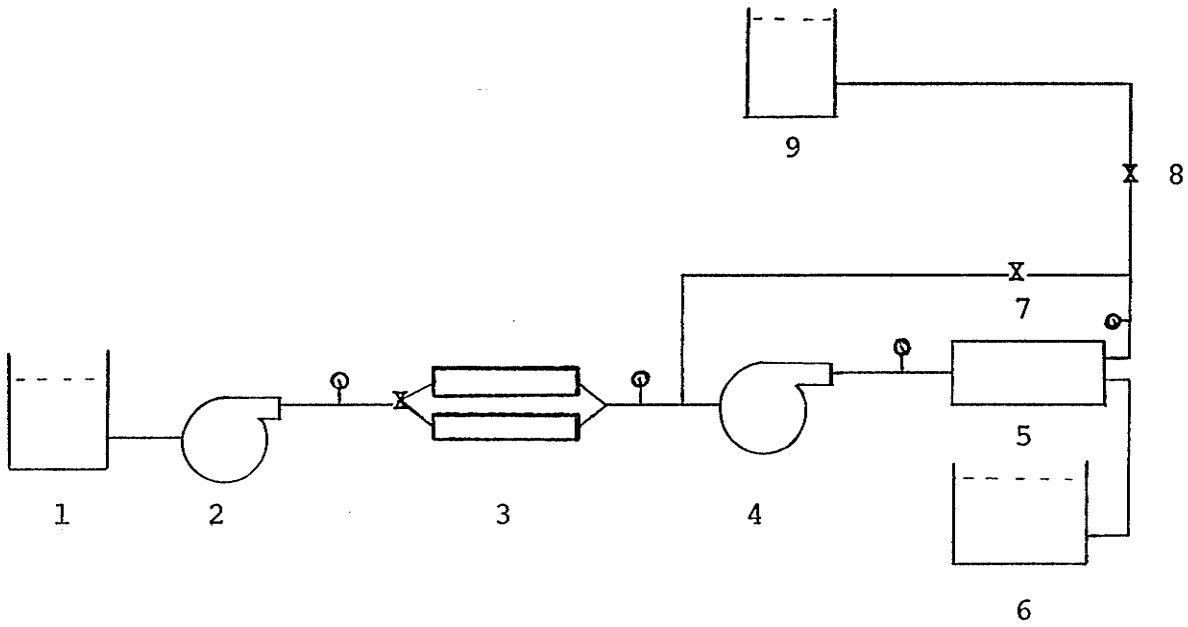


Table 3.1. RO Membrane Specifications

Module Number	Manufacture[1]	Membrane Number	Membrane Type[2]	Membrane Salt Rejection	Membrane Area[3]
192-HR	Osmonics	Sepa-97	CA	Average 97.8% Minimum 96%	1.4 m^2
192-SR	Osmonics	Sepa-92	CA	Average 95% Minimum 92.5%	1.4 m^2
TW30-2026	Filmtec	FT-30	TFC	Average 98% Minimum 96%	1.0 m^2

[1] Osmonics Inc. of Minnetonka, Minnesota
Filmtec Corporation of Minneapolis, Minnesota

[2] CA= cellulose acetate blend
TFC= Thin film composite polyamide

[3] can vary up to 20%

unit via a model Puma 1.5 centrifugal pump supplied by APV Corporation of Chicago, Illinois. A 600 L plastic feedwater tank was supplied by Osmonics. Cooling of the membranes was accomplished using a perforated rubber hose that was wrapped around the pressure vessel and fed with tap water at < 10°C.

3.4.2. Feedwater Collection

Cooling of the protein desludger effluent was necessary to prevent overheating of RO membranes on processing. Effluents collected at approximately 45°C were cooled to about 15°C. Cooling was accomplished by connecting the primary desludger discharge pipe in line to the plate heat exchanger. A 2.5 cm plastic output hose was connected from the heat exchanger to the 600 L feedwater tank. A similar hose was connected from a plant tapwater source to the cooling water section of the heat exchanger. Tap water at 10°C was run through the cooling section as desludger effluent was passed through the sample section. The effluent exited the heat exchanger and filled the feedwater tank at a temperature of 15°C. Approximately one hour was required to fill the feedwater tank using this method.

In the hull fiber effluent trials, no pre-cooling was necessary as the temperature of the effluent was consistently below 10°C. Stainless steel pipe was connected from effluent discharge line of the hull fiber screen directly to the feedwater tank. Collection of 500 L of effluent in the feedwater tank took less than ten minutes.

All effluents processed with the Ajax RO system were prefiltered using in-line filters mounted on the unit. The twinned filter cannister assembly was manufactured by Cuno Engineering Corporation (Meriden, Conn.). Each cannister houses two 23 cm polypropylene filter cartridges. Pressure gauges mounted before and after the filter assembly indicate when replacement is necessary. Filter cartridges were replaced when a 25% drop in feed pressure was recorded.

3.4.3. Processing Method

Figure 3.2. illustrates the flow of this feedwater during processing. Once the feedwater tank has been filled according to the methods outlined in Section 3.4.2., the feed pump was primed with the feedwater effluent. The primed pump was connected to the Ajax RO unit using 2 cm plastic hose. With the RO concentrate valve completely open, the feed pump was turned on. This flushes any existing fresh water from the RO unit. The high pressure pump located on the RO unit was then turned on to pressurize the system. Feed effluent passes from the feed pump through the prefilters and high pressure pump before entering the pressure vessel. Once in the vessel, the effluent is directed into the spiral wound module under pressure. A portion of the effluent in the module passes through the membrane to exit the permeate port as purified water. The remaining feedwaters, now enriched with solutes, may exit the end of the module via the concentrate port or be

recycled to the high pressure module to be processed again. A recycle valve is used to regulate the amount of concentrate blended with feedwater for the adjustment of recovery. The pressure valve regulates the pressure of the system and the permeate production rate. Pressures of 35.2 and 21.1 Kg/cm^2 were used with the Osmonics and Filmtec modules respectively.

A cold water cooling apparatus was installed on the module housing of the Ajax unit to prevent overheating of the RO membranes. An in-line regulating valve served to provide variable temperature control. Temperatures of 25°C and 40°C were used with the Osmonics and Filmtec modules, respectively.

Recovery of the RO system was regulated at approximately 75% using the recycle and pressure control valves. Thus, for every 100 litres of feed effluent processed, approximately 75 liters would be recovered as permeate while 25 liters of concentrate would be produced.

3.4.4. Flow Rate Determination

The flow rates of the RO concentrate and permeate streams were measured every 15 minutes to monitor system recovery. Recovery is calculated according to the equation:

$$\% \text{ Recovery} = \frac{Q_F}{Q_F + Q_E} \times 100$$

where: Q_F = Permeate Flow Rate
Q_C = Concentrate Flow Rate

Concentrate flow was regulated using the pressure valve to obtain a flow rate equal to 25% of the feed flow. This procedure resulted in the achievement of the desired 75% permeate recovery rate which was maintained during all RO trials.

Flow rates were determined by collecting permeate and concentrate waters in separate graduated cylinders over a period of exactly one minute. The flow rates were recorded every 30 minutes in mL/min. Permeate flux was calculated by dividing the permeate flow rate by the area of the membrane contained in the spiral wound module. The Osmonics and Filmtec modules contain approximately 1.4 and 1.0 m^2 of membrane respectively. Flux is expressed in L/m^2 hr.

3.4.5. Sample Analysis

Approximately 250 mL samples of permeate and concentrate water were collected from the RO unit during operation. The first sample was collected after 5 minutes of operation. This allowed any residual fresh water to be flushed from the RO system before sampling. A sample of the effluent feedwater was taken from the feedwater tank just prior to processing. The pH and temperature of the samples were measured and recorded immediately after collection. The samples were then transported to the Food Science Department and refrigerated until analysis. Additional

analysis performed on the samples include:

1) Total Solids
2) Organic loading
3) Carbohydrate
4) Protein (Assay Method)
5) Tannin
6) Ash
7) Chloride

All analyses were performed according to methods outlined in section 3.2.

3.4.6. Initial Module Evaluation

The RO modules on receipt were checked to confirm manufacturer's specifications. Permeate flux and salt rejection were determined according to operating conditions recommended by the manufacturer. Permeate flux was calculated by dividing total permeate flow by the membrane area present in the RO modules used. The procedure used for determining permeate flow is described in section 3.4.4.

Salt rejection was determined using a NaCl solution of known concentration as feedwater. Concentrate and permeate samples were analyzed for salt according to standard methods found in section 3.2. Rejection by the membranes was calculated using the equation:

$$\%Rej = 1 - (C_p/C_{av}) \times 100 = 1 - (C_p/(C_c-C_f) \times 100$$

where: C_{av} = average or mean salt concentration
C_p = concentration of salt in permeate
C_c = concentration of salt in concentrate
C_f = concentration of salt in feed

3.4.7. Membrane Cleaning Procedure

Prior to cleaning, the RO system was flushed with permeate collected during the previous RO trials. This water source was used due to the low level of solutes present and suitable pH and temperature of the water. Low pressures and high flow rates were used during flushing to provide a turbulent cleaning action in the RO module. Flushing was continued for 10 minutes or until the concentrate product water was clear.

RO permeate water was also used for making up membrane cleaning solutions. The strength, pH and temperature of the cleaning solutions were adjusted according to manufacturers directions. The following cleaners were used for membrane cleaning:

Cleaner Name	Type	Manufacturer
1) Ultrazyme 73	enzyme detergent	Osmonics
2) Ultrazyme 93	enzyme detergent	Osmonics
3) NP 20	surfactant	Osmonics
4) NP 23	surfactant	Osmonics
5) Lactonase	enzyme cleaner	----
6) MC-14	alkaline cleaner	Zenon Environ.
7) 2195	acidic resin cleaner	Bird Archer
8) 1% NaOH/.5% SDS	alkaline cleaner	----

The cleaning cycle used was adapted from procedures recommended by the manufacturer of the Osmonics and Filmtec modules. After flushing the RO system with permeate, a 25 L cleaning solution was circulated through the unit under low pressure (210 kg/cm^2) and high flow rate (> 10 L/min). After 15 minutes the unit was turned off to allow a 15 minute soaking period. Circulation of the cleaning solution

was then resumed for an additional 30 minutes. The system was again flushed with fresh water or permeate until the concentrate appeared free of turbidity, color and suds. Measurement of the flow rate was undertaken using fresh water to determine the effectiveness of cleaning. The RO operating conditions used during flow rate measurement were identical to the effluent processing conditions.

3.5. Effluent Recycle Studies

3.5.1. Protein Desludger Effluent Simulation

A laboratory simulation of the Woodstone pea fractionation process was developed over a series of 15 experimental runs undertaken during 1984. The resulting effluents were later used in selected studies. The simulation can be described as follows:

Peas of the Century variety, obtained from Woodstone Foods, were ground to 60 mesh particle size using a laboratory pulverizing mill from Weber Bros. and White Metal Works of Chicago, Illinois. A 15% w/v pea slurry was then produced by mixing 120 g of pea flour with 800 mL of water adjusted to pH 2.5 with concentrated HCl. The slurry was mixed for 20 min at 25^{o}C and centrifuged at 1500 rpm for one minute using a Sorvall bench top centrifuge. This step acts to remove the starch and fiber fraction as a sedimented sludge. The supernatant was then heated to 38^{o}C, adjusted

to pH 4.5 with 10N NaOH and centrifuged at 3000 rpm for one minute to sediment the precipitated proteins. After decanting the supernatant, the protein pellet was resuspended in three volumes of H_2O at pH 4.5 at a temperature of 41°C. An additional centrifugation was performed at 3000 rpm to recover a washed protein and supernatant fraction.

3.5.2. Recycle Study

In step 1 of this study, the above laboratory simulated protein isolation method was used to obtain protein isolates and their respective effluents when primary effluents were recycled into following isolations as feedwater (Fig. 3.3).

In step 2, the secondary effluents from each of the three cycles were combined for use as a starting water for an additional protein isolation cycle. The resulting protein isolate and effluents were tested (section 3.2) for:

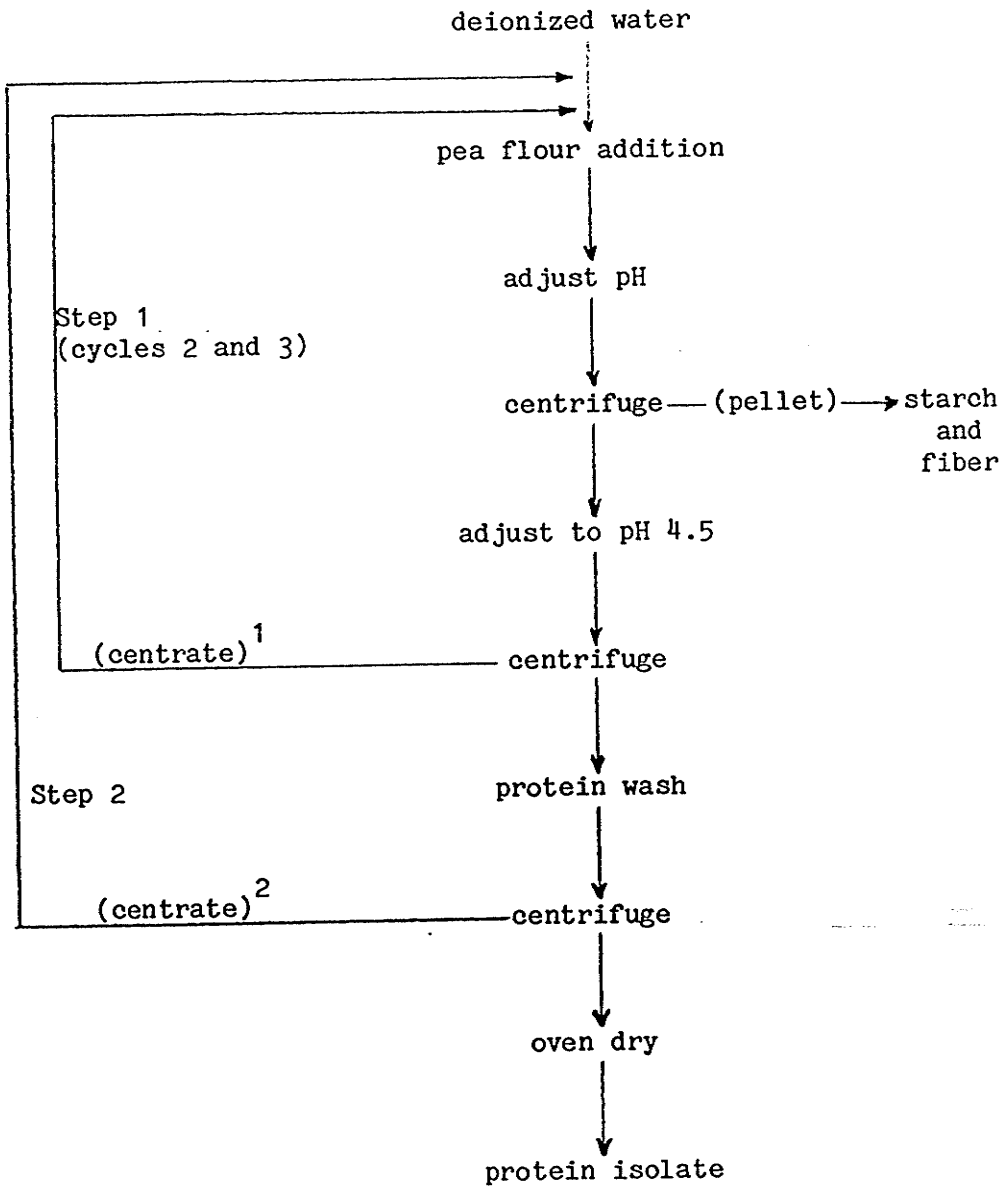
1) Total solids
2) Protein
3) Carbohydrate

3.6. Flocculation Studies

3.6.1. Temperature Induced Floc Study

In this study, effluent from a primary protein desludger (#1) was evaluated for floc development at different temperatures. Approximately 8 L of fresh desludger effluent was vacuum filtered using Whatman 934-AH paper to remove all suspended matter. The effluent filtrate

Figure 3.3. Recycle scheme.



[1]primary effluent equivalent

[2]secondary effluent equivalent

was immediately divided into 1 L aliquots and placed on Corning hot plates with stirrers. One aliquot was heated at each temperature of 35°, 45°, 60°, 70°, 80° and 90°C with stirring. Two samples were placed in the refrigerator at approximately 4°C. All temperatures were monitored using standard mercury bulb thermometers. One refrigerated and all of the heated samples were held at their respective temperature for exactly one hour before cooling to room temperature. The second refrigerated sample was held at 4°C for 24 hours before analysis.

Samples showing no sign of floc formation (4°, 35°, 45°C) were again vacuum filtered on 934-AH paper for the quantitative determination of suspended solids. All other samples were centrifuged to obtain a floc pellet. The pellet was freeze dried, oven dried and weighed prior to analysis for protein and ash content.

Supernatants from the second and third centrifugations correspond to the primary and secondary protein desludgers from the Woodstone process, respectively. These desludger effluent counterparts were analyzed for the following parameters according to the Standard Methods found in section 3.1..

1) Total solids
2) Organic loading (COD)
3) Carbohydrates
4) Total nitrogen
5) Tannin

3.6.2. $FeCl_3$ Induced Floc Study

In this study, 2M $FeCl_3$ was added to 100 mL aliquots of prefiltered primary desludger effluents in amounts ranging from 0 to 10 mL. The samples were stirred and held for one hour at 40°C in a temperature controlled water bath. The effluent samples were then refiltered on pre-weighed, dessicated Whatman 934-AH paper to remove any flocculated material. Floc formation was determined gravimetrically as suspended solids remaining on the filter dried at 103°C .

3.7. Antifoam Treatment Studies

3.7.1. Activated Carbon Treatment

In this study, powdered and granular activated carbon were added to 250 mL aliquots of primary desludger effluent at dose rates of 0.0 to 0.50 g/100 mL. After mixing, the solution was filtered through Whatman 934-AH paper to remove the carbon. A 100 mL portion of each filtrate was poured into graduated cylinders and then shaken vigoursly for ten seconds. The amount of foam produced was measured in milliliters.

3.7.2. Corning Antifoam Treatment

In this study, Corning FG-10 Antifoam was added to 100 mL aliquots of primary desludger effluent at levels of 0.0 to 0.5 mL/100 mL. The solutions were then poured into graduated cylinders, shaken, and measured for foam capacity.

4. RESULTS AND DISCUSSION

4.1. Effluent Characterization

Woodstone Foods is presently the only plant in North America using a wet process for the isolation of field pea components. In full operation, this plant may use in the order of 600,000 L of fresh water per day. A correspondingly large discharge of process effluent results in considerable municipal treatment charges as well as environmental burden. A potential for in-plant water reuse/recycle of these process effluents exists. The literature is, however, void of references concerning the actual data for this wet process. Therefore, a series of studies were undertaken to characterize the effluent streams resulting from this unique process. The results of this work would be beneficial in determining the feasibility of in-plant treatment for the purpose of water recycle. Both a random sample and a multiple sampling scheme were used to characterize the process effluents.

4.1.1. Random Sample Study

Over a six month interval, a series of four sets of effluent samples were collected from the sources listed in section 3.1.1. These samples were collected as random samples during the morning operation of the plant, on varied dates. The analytical results of the eight effluent sources examined are presented in Table 4.1..

TABLE 4.1. Characterization of Field Pea Process Effluents[1]

Effluent Source	Total Solids (mg/L)	Suspended Solids (mg/L)	COD (mg/L)	Carbohydrate (mg/L)	Total Nitrogen (mg/L)	Tannin[2] (mg/L)	pH
Pea Wash	5410 ±995	2845 ±1080	4940 ±2542	2170 ±667	279 ±41	40 ±14	5.84 ±1.59
Fiber Decanter	830 ±356	155 ±100	1160 ±433	217 ±222	69 ±3	33 ±29	6.50 ±0.67
Starch Decanter	5070 ±1484	1865 ±148	5178 ±1472	1800 ±561	288 ±46	45 ±22	6.21 ±0.92
1' Desludger #1	18600 ±2812	1313 ±823	19400 ±6047	8970 ±2244	726 ±232	152 ±46	4.86 ±0.39
1' Desludger #2	18960 ±5458	937 ±892	20600 ±5751	8870 ±2169	772 ±253	146 ±39	4.86 ±0.45
2' Desludger #1	5090 ±843	190 ±106	5420 ±2515	1630 ±603	296 ±64	43 ±13	4.77 ±0.34
2' Desludger #2	4930 ±789	105 ± 75	5100 ±2499	2040 ±665	198 ±17	48 ±14	4.56 ±0.71
Outside Tank	11350 ±2965	1650 ±361	11400 ±1687	2500 ±1951	538 ±239	116 ±35	6.64 ±1.52

[1]Means from four sampling periods
[2]Tannin refers to the group of naturally occuring phenolic compounds comprised mainly of flavonoids

The mean pH of the desludger effluents, varied from 4.56 to 4.86. These values deviated from the optimum pH of 4.5 used in the protein precipitation process. Variation from optimum conditions may have decreased the efficiency of the process and increased the level of soluble protein found in the resulting effluents (Nickel, 1984).

The measure of total solids present in the effluent could be used as an indicator of system efficiency and product loss. Totals solid means in the effluents varied from 830 mg/L to approximately 19,000 mg/L for the fiber decanters and primary protein desludgers, respectively in the membrane equipment. The effluents containing low levels of total solids may be suitable for reuse in plant processes, with minimal or no pretreatment. Effluents contributing the largest concentrations of total solids, to the plant discharge, may be suitable for reuse with minimal or more extensive treatment.

The suspended solids means varied from 105 mg/L for the secondary desludger effluent to 2845 mg/L for the pea wash water. The high level of suspended solids in the pea wash can be attributed to residual plant matter (pea hulls, pod fragments) as well as dirt and other foreign material found in the raw peas.

The total organic matter content of the effluents, as measured by the chemical oxygen demand (COD) test, varied considerably between sources. COD means ranged from

1160 mg/L to 20,600 mg/L for the fiber decanter and primary desludgers, respectively. This parameter indicated the loss of organic constituents to the discharged process waters. Losses of soluble and insoluble organic matter decrease product yield and increase municipal treatment charges. The recycle/reuse of these effluent waters may decrease the solubilization effect of soluble components during processing. In addition, an increase in the insoluble component recovery level should be experienced on reuse of selected effluent streams. This could economically benefit the plant.

Organic matter present in the effluent streams was further characterized as carbohydrates, total nitrogen and tannin content. Carbohydrate was present, as the major component, in all effluents tested. Data means varied from 217 mg/L for the fiber decanter effluent to approximately 9000 mg/L for the primary desludgers. This component accounted for 26 to 48% of the total solids measured, respectively. A complex mixture of simple sugars, oligosaccharides and high molecular weight polysaccharides such as starch, gums and pectin make up the carbohydrate portion of these waters.

Total nitrogen means varied from 69 mg/L for the fiber decanter to 772 mg/L for the primary desludgers. It was important to determine the base level of protein lost in the desludger effluents; an increase would indicate the system was not operating at maximum efficiency. Subsequent work

indicated that a major portion of the nitrogen present in these waters were non-protein nitrogen in nature.

Mean tannin values of 33 to 152 mg/L were recorded in the effluent samples tested. The tannins were measured in this study, since high concentrations could cause undesirable protein precipitating reactions in the membrane equipment. Also, the presence of high levels of tannins could considerably reduce the sensory quality of water and food products. Tannins may impart bitter flavors, off odors and color to food and water (How and Morr, 1982).

The analyses undertaken in this study offer an approximation of the soluble and insoluble component concentrations, present in the plant process streams. Mean component concentrations varied considerably between effluent sources. These results indicated that effluent streams such as the fiber and starch decanter were low in total solids and may not require additional treatment prior to plant process recyclization. The reuse of effluents with varied organic matter contents may be beneficial in improving process efficiency and product recovery.

Composition means obtained from outside tank effluent analysis can be used as an estimate of the total mean plant discharge, as this source is the composite of all plant waters. This effluent source is tested by municipal authorities when sewage surcharges are levied. The City of Winnipeg Sewer By-Law No. 505/73 enforces regulations

similar to those enforced by the City of Portage la Prairie. This by-law states that the organic matter content of discharged waters must be below 300 ppm (300 mg/L) as measured by biochemical oxygen demand (BOD). The mean discharge of organic matter measured in the composite effluent from the outside tank was 11,400 mg/L. Although, previous work has shown that the BOD measurements of organic material, in industrial wastes, were approximately 50% lower than COD measurements, considerable violation of the maximum level is still evident.

Suspended solids is another parameter used to determine sewage surcharges. The Sewer By-Law allows for a maximum level of 350 ppm (350 mg/L) suspended solids, as determined in accordance with "Standard Methods". The mean concentration of suspended solids present in the plant composite discharge was 1650 mg/L. This parameter was also in violation of the allowed limits. Primary filtration of any effluents leaving the plant would reduce the concentration of suspended solids to an acceptable level.

Section 4.9 of the Sewer By-Law lists pH levels below 5.5 and above 9.0 as a prohibited substance subject to surcharge. The composite effluent at the plant was well within the allowable limits with a mean pH of 6.64.

4.1.2. Multiple Sampling Study

An expanded characterization study was undertaken to

determine the compositional variation between the primary and secondary protein desludger effluents. The desludger sources were chosen because of their high demand on fresh water and subsequent discharge of large volumes (approximately 400,000 L/day) of highly loaded effluent. The multiple sampling plan consisted of collecting desludger effluents on an hourly basis for a six hour period. Two sets of samples were collected in a period of a month. Section 3.1.3. lists the analyses performed on each sample collected.

The results of the effluent analysis is provided in Table 4.2. Considerable deviation from the optimum processing parameters of pH 4.5 and temperature of 45^{o}C was noted. Process temperatures ranged from 38.0^{o} to 50.3^{o}C and pH varied from 4.10 to 4.72 during these trials. This deviation could introduce significant variability in effluent composition and process efficiency.

Total solids, COD, carbohydrate, total nitrogen and tannin analysis means were similar to data obtained from the random sampling plan. Carbohydrate was the major component in the effluents accounting for 42-47% of the total solids. Protein (protein nitrogen x 6.25) accounted for 8-12% of the solids. Component concentrations in the primary desludger effluents were approximately 3 to 4X that found in secondary desludger effluents. This difference was expected as the secondary desludgers acted only to wash the protein

TABLE 4.2. Protein Desludger Effluent Characterization[1]

Effluent Source	Total Solids (mg/L)	COD (mg/L)	Carbohydrate (mg/L)	Total Nitrogen (mg/L)	Protein Nitrogen (mg/L)	Tannin (mg/L)	pH
1' Desludger #1	20680 ±1660	18900 ±1521	8430 ±989	837 ±46	349 ±21	156 ±18	4.47 ±0.17
1' Desludger #2	21970 ±2071	19500 ±1816	8530 ±1084	869 ±60	282 ±19	159 ±15	4.47 ±0.16
2' Desludger #1	5914 ±1656	6370 ±1428	2890 ±1263	256 ±18	78 ±13	50 ±16	4.48 ±0.17
2' Desludger #2	6723 ±1629	5800 ±2409	2890 ±1058	249 ±22	72 ±8	49 ±16	4.48 ±0.18

[1]Data is the mean of 12 observations

isolate with fresh water, while the primary desludgers discharged waters remaining from the pea grind, fiber, starch and protein isolation processes.

Protein nitrogen accounted for approximately 31 % of the total nitrogen present in the primary and secondary effluents (Table 4.2.). Mean values of 1970 and 470 mg/L of protein (protein nitrogen x 6.25) were found in these waters respectively. Thus, the majority of the nitrogen found in these effluents represent amino acids, peptides, nitrates and other non-protein nitrogenous compounds.

For most of the parameters tested, the standard deviation was large. This variation was the result of fluctuating process conditions which may have resulted in variable product composition.

4.2. Ultrafiltration Studies

4.2.1. Membrane Types

Amicon manufactures several UF modules ranging from 1,000 MWC to 100,000 MWC. All modules contain polysulfone based hollow fiber membrane. Recommended uses of the available modules and concentrator/dialyzer unit include the processing of solutions containing proteins, enzymes, extracts and colloidal products. No other manufacturer produces UF modules that can be used with the Amicon DC-2 UF system used in this study.

As the recovery of the protein fraction was a major objective of this study, membrane cutoffs were chosen that would retain the protein and allow removal of low molecular weight components. As suitable 10,000 and 30,000 MWC modules were available in the Food Science Department, their use was incorporated into these studies. The operating parameters of the UF trials are presented in Table 4.3.

The use of a 50,000 MWC membrane was requested by the plant management to provide increased permeate production rates. Increased initial permeate flux can normally be expected, with increasing pore size, from membranes made with identical materials. This membrane was obtained from Amicon.

4.2.2 Feedwater Source

Primary protein desludger effluents were used as the feedwater source for the UF and following RO experiments. This effluent source was the major contributor to organic loading in the plant process. In addition to effluent purification, the company wished to determine the feasibility of by-product recovery from effluents using membrane processing. A commercial application of UF would also act to recovery the membrane permeate for potential recycle in preceeding processes. Characterization of the UF feedwaters and fractions are shown in Tables 4.4. to 4.10. Approximately 200 L of primary desludger effluent was collected in a tank to provide a representative feed sample.

TABLE 4.3. Operating Parameters of UF Trials[1]

Membrane MWC[2]	Concentration Factor	Effluent pH	Effluent Temperature
10,000	10X	4.47	46.0°C
30,000	10X	4.42	46.0°C
50,000	10X	4.66	43.5°C
30,000	20X	4.38	30.0°C
50,000	20X	4.50	42.0°C
Activated Carbon Pretreatment			
30,000	20X	4.57	55.0°C
50,000	20X	4.29	48.0°C

[1]Protein desludger effluent as feedwater source
[2]Molecular Weight Cut-off

TABLE 4.4. COD Analysis of UF Fractions of Desludger Effluent(mg/L)

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	19500	19400	22600	19200	21000
Concentrate	70600	78000	54200	83100	72500
Permeate	12400	11500	16100	13500	13000
Rejection	72.5%	76.4%	58.1%	86.8%	72.2%

TABLE 4.5. Total Solids Analysis of UF Fractions of Desludger Effluent(mg/L)

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	21300	20680	23800	20890	23180
Concentrate	59440	64620	55120	78060	77760
Permeate	15600	14580	19450	16650	16980
Rejection	61.4%	65.8%	50.7%	66.3%	66.4%

TABLE 4.6. Carbohydrate Content of UF Fractions of Desludger Effluent(mg/L)[1]

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	8720	9310	11200	11400	11900
Concentrate	26900	25000	30300	42300	41900
Permeate	6540	5370	8340	8670	8500
Rejection	63.3%	68.7%	60.0%	67.7%	68.4%

[1]Calculated by difference

TABLE 4.7. Total Nitrogen Analysis of UF Fractions of Desludger Effluent(mg/L)

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	940	910	1050	830	970
Concentrate	4100	4460	2910	5010	4930
Permeate	470	480	760	450	530
Rejection	81.0%	82.1%	61.6%	84.6%	82.0%

TABLE 4.8. Protein Analysis of UF Fractions of Desludger Effluent(mg/L)[1]

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	1910	1880	2090	1680	274
Concentrate	14600	17100	11300	19600	18800
Permeate	35	125	925	111	94
Rejection	99.6	98.7%	86.2%	99.0%	99.0%

[1]Protein = TCA Precipitable NH_3-N x 6.25

TABLE 4.9. Tannin Analysis of UF Fractions of Desludger Effluent(mg/L)

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	155	154	184	125	144
Concentrate	773	775	523	725	845
Permeate	71	71	139	67	88
Rejection	84.7%	92.4%	60.7%	84.2%	91.1%

TABLE 4.10. Ash Analysis of UF Fractions of Desludger Effluent(mg/L)

Concentration Factor	10X	10X	10X	20X	20X
Trial	10000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Feedwater	-	-	5993	4339	5258
Concentrate	-	-	6640	4445	5015
Permeate	-	-	6260	4752	5166
Rejection	-	-	0.9%	0.0	0.0

The temperature and pH of the effluents were recorded before use in the UF trials. Table 4.3. shows that the pH and temperature of the feedwater samples varied considerably. The pH ranged from 4.29 to 4.66, while the temperature ranged from 30° to 55°C at the time of collection. Optimum process conditions established by the plant, were pH 4.5 and temperature 40-45°C. Variations in pH, as little as 0.1 pH unit, could effect the protein yield in the precipitation step. This could result in higher or lower soluble protein concentrations in the effluent. Increased levels of protein could overload an operational membrane system, whereas, decreased levels may negate the purpose of the system for by-product recovery. Similarly, difficulties may occur with other organic and inorganic constituents due to decreased solubility or membrane rejection properties at various pH levels. A reduction in component solubility, due to precipitation or crystallization, may cause excessive membrane fouling (Kuo and Cheryan, 1983).

Temperature variations also influence process efficiency and the resulting effluent composition. Temperature control is necessary during membrane processing to regulate permeate production rates. As temperature increases from 0° to 50°C, permeate production also increases. In addition, operating temperatures over 50°C can cause damage to polysulfone membranes (Anon., 1984b). This is a concern as effluent temperatures as high as 55°C

were experienced during the course of sample collection.

4.2.3. Ultrafiltration Fraction Analysis

4.2.3.1. Fraction Composition

The 10,000, 30,000 and 50,000 MWC modules were used in the 10X concentration trials, while only the latter two modules were used in the 20X concentration trials (Tables 4.4. to 4.10.). Use of the 10,000 MWC module was discontinued in the 20X concentration trials as the protein rejection rates were found to be similar to the 30,000 MWC module, with considerably reduced initial premeate flux. High permeate flux is necessary to optimize a high capacity UF plant for water treatment. A 20X volume concentration was used in an attempt to obtain a UF concentrate with a higher total solids content than that provided by the 10X volume reduction trials. A higher level of total solids is desirable if the concentrate fraction is to be dried for use as a food or feed by-product.

The feedwaters and UF fractions were analyzed for the parameters listed in section 3.3.4. The COD levels in the concentrate fractions ranged from 54,200 mg/L to 83,100 mg/L. This represented a 3-4X concentration of organic constituents found in the effluent feedwater. Organic loadings of the permeates remained relatively high at 11,500 - 16,100 mg/L. Concentration factors of 10X and 20X produced mean total solids values of 57,700 and

77,900 mg/L in the concentrate, respectively. Thus, only a small portion of the constituents in the feedwater must have possessed a molecular weight large enough to be retained by the UF membranes. This observation was confirmed by the relatively high concentration of organic and total solids remaining in the permeate water, following the UF processing.

Approximately 52-76% of the carbohydrate found in the feedwater samples passed through the membrane to the permeate side. This suggested that the feedwater contained a considerable level of low molecular weight mono, oligo and polysaccharides. Carbohydrates composed 40-55% of the solids in the UF concentrate. This high concentration may be responsible for the gelling of the concentrate fraction that was experienced upon cooling after UF treatment. This phenomenon could have caused membrane fouling during processing, if a drop in feedwater temperature was experienced.

From Tables 4.7. and 4.8., calculations reveal that approximately 30% of the Kjeldahl N present in the feedwater is of protein origin. Table 4.8. also shows that the mean protein rejection rates were 98.4%, 94.1% and 77.2% for the 10,000, 30,000 and 50,000 MWC UF membranes used, respectively. A high level of protein rejection is necessary if UF is to be used as an effective method of protein recovery. Overall, protein constitutes less than 0.2% of the feedwater effluent, and approximately 8.0% of

the total solids on a dry weight basis. These low values suggest that it may not be practical to recover a protein by-product. Additional benefits of membrane processing may be realized if other high value minor components can be recovered.

Tannin was concentrated approximately 4X using the UF membranes (Table 4.9.). This level increase in the concentrate fraction could result in a phenol-protein reaction; ultimately leading to precipitation. Pretreatment of the effluent may be necessary to prevent continued fouling of the fibers. Excessive fouling of the membranes creates difficulties upon scaling-up to an industrial size process.

It should be noted that no increase in the ash content was found upon concentration of the feedwater (Table 4.10.). This was expected as the molecular weight of the inorganic constituents, such as free ions and salts, are much lower than the MWC of the membrane used. Also, the electrostatic repulsive forces between ions and membrane surfaces are negated when the membrane pore size is larger than the adjacent monolayer of water (Sourirajan and Matsuura, 1985).

4.2.3.2. Effect of PAC Pretreatment on UF Fraction Composition

A study was undertaken to evaluate the effectiveness of powdered activated carbon (PAC) for effluent pretreatment.

A reduction of tannin-like phenolic components present in the effluents was anticipated using PAC pretreatment. This would decrease the possibility of phenol-protein interaction thought to cause membrane fouling. A carbon dosage of 1g/L of wastewater is generally recommended by activated carbon companies for pretreatment purposes. Levels higher than 1g/L may not be economically feasible.

The analysis results of the UF fractions from PAC treated effluent, as UF feedwater, and untreated protein desludger effluent, as the control, are shown in Tables 4.11.(a) and 4.11.(b). All fraction parameters tested, were similar in concentration to that of the non-PAC treated trials (Tables 4.4. to 4.10.). Rejection of protein nitrogen was greater than 98%, while total solids, carbohydrate, total nitrogen and tannin fell within a range of 66-85%. Rejection of the ash component was approximately 0%. This low value is characteristic of most types of UF membranes. Rejection patterns between the 30,000 MWC and 50,000 MWC membranes were very similar (Tables 4.11.(a) and 4.11.(b)). This similarity may be explained by considering two factors. First, the molecular weight exclusion limit of UF membranes is generally not very sharp. Secondly, the mixture of components in the effluent ranges widely from monovalent ions to macromolecular colloids . These factors would illustrate the inability of the membranes to provide a distinguishable rejection performance between the 30,000 and 50,000 MWC trials.

TABLE 4.11.(a). Analysis of UF Fractions from Activated Carbon Pretreated Desludger Effluent

Trial	30000 MWC	50000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Parameter	COD (mg/L)		Total Solids (mg/L)		Carbohydrate[1] (mg/L)	
Control	19700	15400	21990	17650	11500	9100
PAC	19200	15000	21850	17350	11300	9200
Conc.	92100	66400	81810	68520	48400	35000
Perm.	13900	10600	17450	13840	9720	7030
Rejection	75.0%	74.0%	66.3%	67.4%	67.4%	68.2%

[1]Calculated by difference

Table 4.11.(b). Analysis of UF Fractions from Activated Carbon Pretreated Desludger Effluent

Trial	30000 MWC	50000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC	30000 MWC	50000 MWC
Parameter	Total Nitrogen (mg/L)		Protein (mg/L)		Tannin (mg/L)		Ash (mg/L)	
Control	900	750	1794	1219	163	98	4910	3856
PAC	890	670	1731	1181	151	84	4992	3934
Conc.	4650	4780	18380	16690	642	633	4340	3700
Perm.	430	470	169	156	105	52	5040	3876
Rejection	84.5%	82.8%	98.3%	98.2%	73.5%	85.5%	0.0%	0.0%

Reductions in COD, total solids, total and protein nitrogen and tannin were noted in the PAC-treated feedwaters. Mean component losses of less than 10% occurred from the PAC treatment at the dosage used. This was beneficial, as activated carbon was not used to remove the effluent constituents which could be concentrated for recovery by ultrafiltration processing.

As the level of protein and tannin-like phenolic compounds remained relatively constant, a PAC treatment would not be useful in the reduction of phenol-protein interaction thought to contribute to membrane fouling.

An increase in ash of approximately 5% was noted after treatment of the effluent with PAC (Table 4.11.(b)). This slight increase may be due to carbon contamination during filtration. A mass balance of all UF trials is provided in Appendix 2.

4.2.3.3. Qualitative Analysis

The primary desludger effluents used in the ultrafiltration trials were yellow in color, slight to moderate in turbidity, with a marked stale-pea odor. The resulting permeate fractions possessed the characteristic yellow color and some odor, but appeared free of turbidity. The PAC-treated effluents were noticeably reduced in color and turbidity. The color of the permeate from these trials was less intense than in the non PAC-treated trials.

Permeates of this quality may be useful for reuse in some plant processes.

Concentrates produced from these trials appeared off-white in color, viscous and tacky in consistency. In the 20X concentration trials, the resulting concentrate gelled upon cooling. This phenomenon may be attributed to the concentration of amylose and other high molecular weight carbohydrates liberated from the peas during processing. Gelling of UF concentrates could cause membrane fouling problems if a drop in operating temperature is encountered.

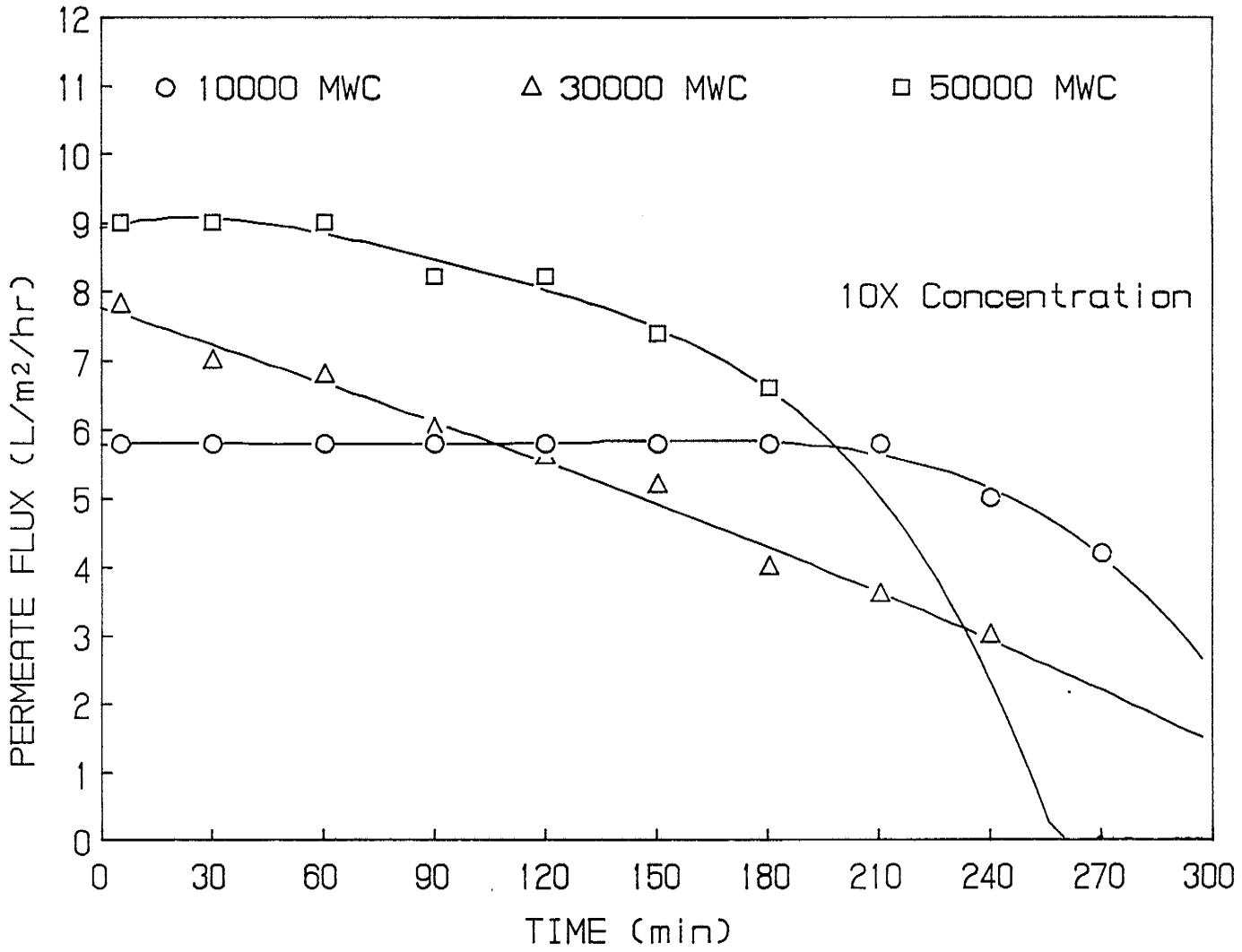
Application of a 1% iodine solution to the UF feedwaters and concentrates produced an intense blue-black coloration. The same solution mixed with UF permeate produced no change in color. Thus, the UF permeate must be relatively free of amylose.

4.2.4. Ultrafiltration Permeate Flux

4.2.4.1. Coarse Prefiltration

The flux values obtained using the Amicon 10,000, 30,000 and 50,000 MWC hollow fiber modules, with coarse prefiltration of desludger effluent feedwaters, is illustrated in Fig. 4.1. The trials were terminated when a 10X volume concentration was reached. As the rate of flux varied between membranes, the time required to reach a 10X volume concentration also varied. Increased initial flux was apparent with increasing molecular weight cutoff. Loss

Figure 4.1. Effect of time on UF permeate flux with coarse prefiltration.


12
11
10
9
8
7
6
5
4
3
2
1
0
PERMEATE FLUX (L/m²/hr)
○ 10000 MWC
△ 30000 MWC
□ 50000 MWC
10X Concentration
0
30
60
90
120
150
180
210
240
270
300
TIME (min)

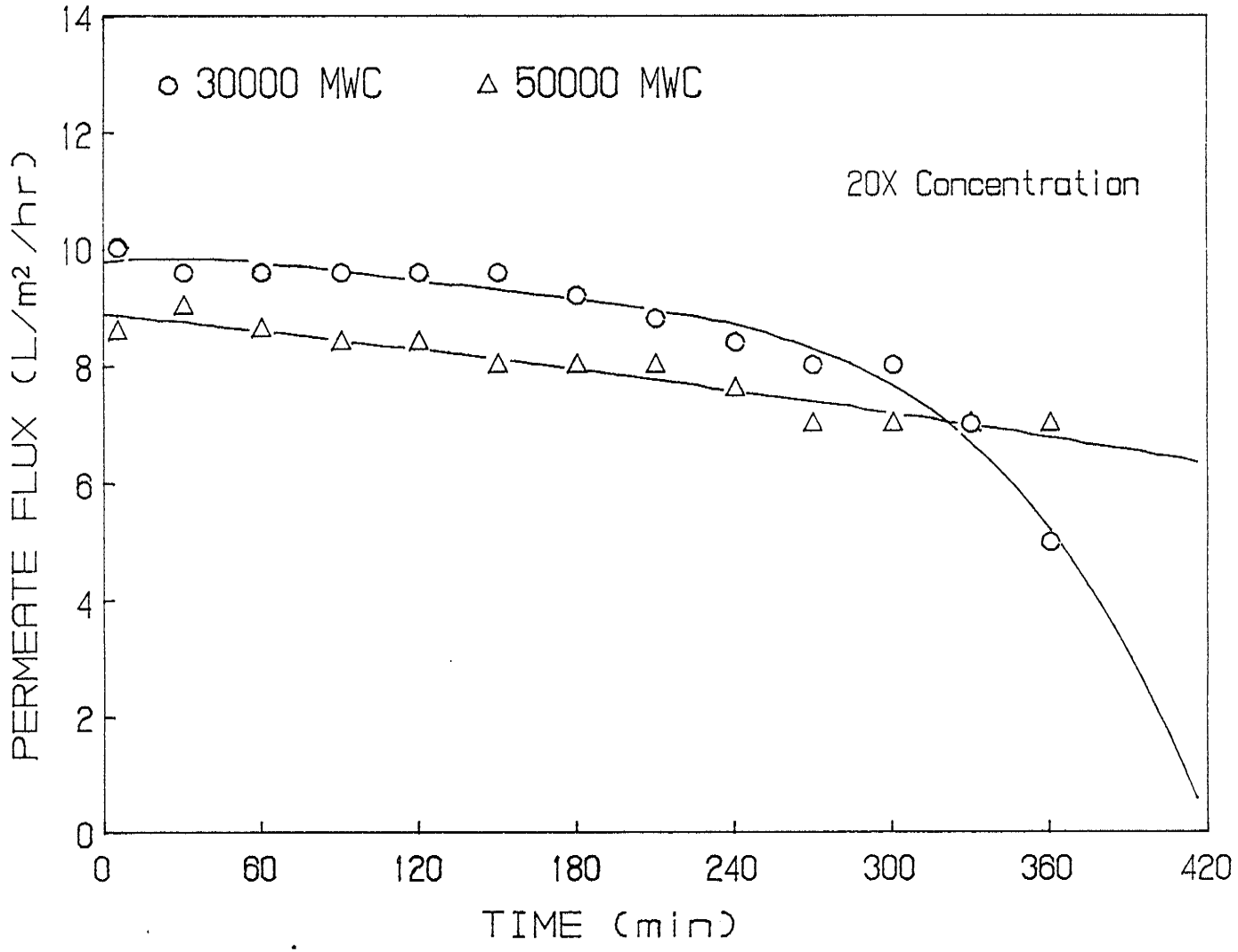
in flux over the concentration period was 17, 62 and 27% for the 10,000, 30,000 and 50,000 MWC membranes, respectively. The wide range in flux loss values indicated that membrane fouling may be a function of pore size. Pores existing in the membrane of the 30,000 MWC module may have become clogged by a component species with a given molecular weight. However, this species may have passed through the larger pores of the 50,000 MWC module and have been totally occluded by the smaller pores of the 10,000 MWC module.

Prefiltration of the feedwater effluent was accomplished using a filter supplied by Amicon Inc. The reuseable filter was manufactured for use in the Amicon UF unit, but was not micron rated for particle retention. Therefore, this filter may be less effective than conventional 0.5 to 5 paper filters used for the removal of suspended solids. High levels of suspended solids may contribute to membrane fouling, and are therefore, not desirable.

4.2.4.2. Whatman #5 Pre-filtration

The effect of micron prefiltration on the flux values obtained using the Amicon 30,000 and 50,000 MWC hollow fiber modules is illustrated in Fig. 4.2. These trials were terminated when a 20X volume concentration was reached. A 20X concentration factor was used to produce a concentrate fraction with increased total solids concentration for by-product recovery. Use of the 10,0000 MWC UF module was

Figure 4.2. Effect of time on UF permeate flux with Whatman #5 prefiltration.


o 30000 MWC
Δ 50000 MWC
20X Concentration
PERMEATE FLUX (L/m² /hr)
TIME (min)
0
2
4
6
8
10
12
14
0
60
120
180
240
300
360
420

discontinued in favor of modules with higher initial permeate flux.

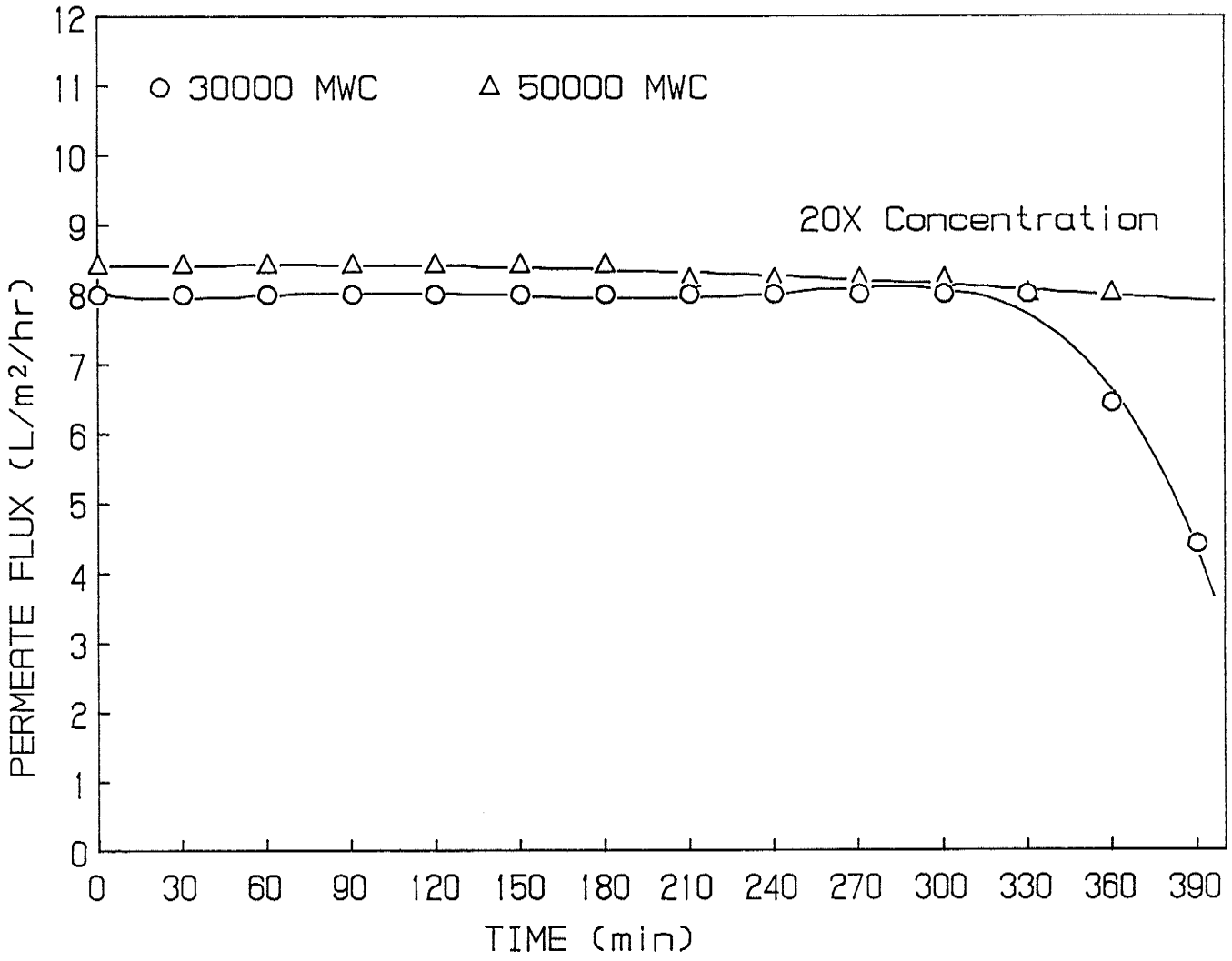
Loss in flux over the period of concentration was 40 and 5% for the 30,000 and 50,000 MWC modules, respectively. These flux values were considerably improved over those obtained in previous 10X concentration trials. This indicated that prefiltration of feedwater effluents with Whatman #5 filter paper decreases membrane fouling. Fine particle occlusion properties can be expected from this paper as it has a 2.5 micron particle retention rating. Decreased levels of permeate flux were anticipated due to the increased time of UF operation and higher total solid levels encountered with a 20X volume concentration.

4.2.4.3. Activated Carbon and Whatman #5 Pre-treatment

The effect of PAC and Whatman #5 pretreatment on UF permeate flux values, using desludger effluent as feedwater, is shown in Fig. 4.3. The 30,000 and 50,000 MWC modules were used to concentrate 2 L of effluent 20 times. A standard PAC dosage of 0.1% (w/v) was used in order to approximate a maximal commercial treatment.

Permeate flux was found to decrease by 50% using the 30,000 MWC module. This value was similar to the 45% flux loss obtained in previous trials using Whatman filtered effluent with no PAC treatment. The 50,000 MWC trial shows a less than 10% reduction in flux over the period of three

Figure 4.3. Effect of time of UF permeate flux with PAC and Whatman #5 pretreatment.


12
11
10
9
8
7
6
5
4
3
2
1
0
○ 30000 MWC
△ 50000 MWC
20X Concentration
PERMEATE FLUX (L/m²/hr)
0
30
60
90
120
150
180
210
240
270
300
330
360
390
TIME (min)

hours. This contrasts with the results of the previous filtration trial and may be due to the difference in composition of the effluent feedwater. This point emphasizes the need for consistent feedwater composition and careful monitoring of plant processing conditions.

The activated carbon appeared to have limited effect on flux improvement in both trials performed, as final flux values were very similar to the non PAC-treated trials, and visual fouling of the hollow fibers was evident (Fig. 4.4.). The fouling layer was formed in the lumen of the hollow fibers and could be phyically removed for examination. Staining of the these tubes with Ponceau red dye indicated that very little protein was incorporated into these structures. However, the application of a dilute iodine solution resulted in dark staining demonstrating the considerable amount of amylose present (Fig. 4.5.).

4.2.5. Ultrafiltration Trial Summary

The protein desludger effluents used in these trials were found to vary considerably in pH, temperature and chemical composition. Fluctuating feedwater parameters may alter the performance or cause fouling difficulties on membrane processing scale-up. Steps should be taken to obtain the most consistent feedwater source possible.

Permeate produced from UF of effluents was found to possess relatively high levels of residual organic

Figure 4.4. Fouling layers from the UF hollow fibers.

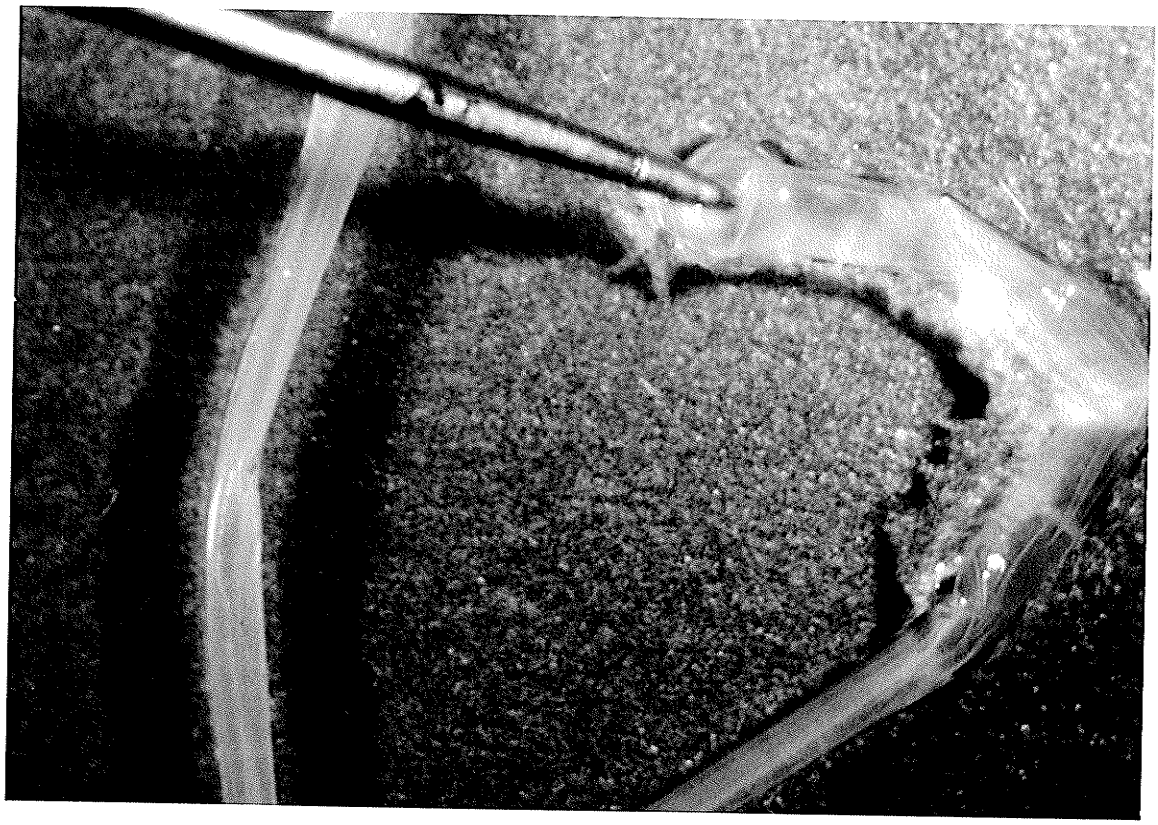
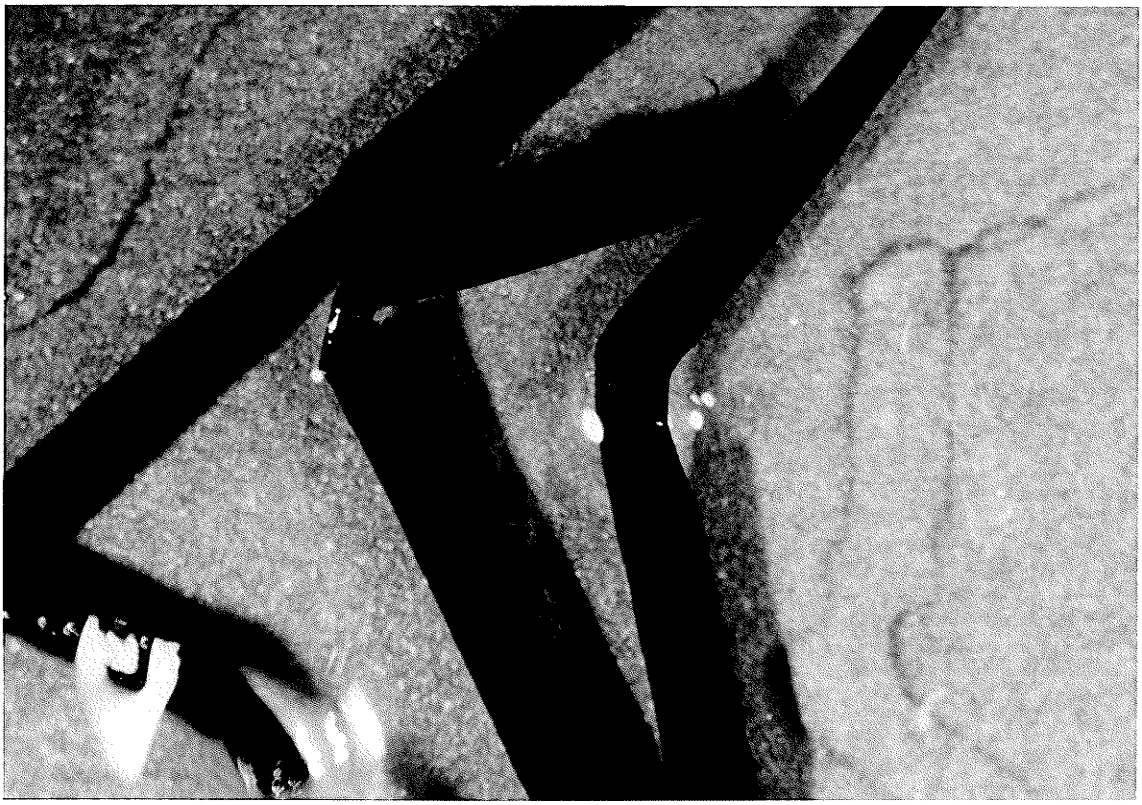


Figure 4.5. Iodine reaction to UF fouling layers.



components and total solids. This indicated that UF alone was not effective in removing the components present in these effluents. The concentrate produced by UF contained low levels of protein and high levels of carbohydrate, suggesting that recovery of a high purity protein by-product was not feasible. However, the recovery of other high value minor components may make UF of these waters economically feasible.

Pretreatment of effluents using fine filtration was found to reduce membrane fouling. The use of PAC pretreatment did not produce beneficial effects in the prevention of membrane fouling, but did improve the color and level of turbidity present in the UF feedwater and the resulting fractions.

4.3. Filmtec Nanofiltration Membrane Trial

This membrane possesses rejection characteristics intermediate of RO and UF. In this experiment Woodstone's fiber isolation effluent was chosen to evaluate Filmtec's NF-40 nanofilm membrane. A batch mode of concentration was utilized to process 200 L of prefiltered effluent to 150 L of permeate and 50 L of retentate.

The analysis results of the original plant effluent, pre-filtered effluent and resulting concentrate and permeate fractions are shown in Table 4.12. A 57% reduction in total solids occurred due to prefiltration of the effluent with

TABLE 4.12. Analysis of NF-40 Fractions of Fiber Decanter Effluent

Sample	Time (min)	Total Solids (mg/L)	Protein (mg/L)	Carbohydrate[1] (mg/L)	Ash (mg/L)	Tannin (mg/L)
Control	–	3752	879	2290	584	57
Filtered Feed	0	1592	38	1030	524	43
Concentrate	5	1680	76	1040	568	54
	30	3220	95	2130	996	74
	60	4728	143	3210	1376	118
Permeate	5	196	<10	10	176	4
	30	304	<10	42	252	5
	60	432	<10	142	280	6

[1]Calculated by difference

filter pads and filter-aid. The protein content was reduced by 96%, while carbohydrate and ash were reduced by 55 and 10% respectively. This was anticipated, as previous studies had shown that approximately half of the total solids found in this effluent source were present as suspended solids.

The results indicate that the concentration of total solids in the feedwater increased approximately 3X during processing. This value was lower than the 4X volume concentration factor used for processing the effluent. The difference in values was likely due to the passage of some low molecular weight components through the 400 dalton MWC membrane. In addition, some solutes may have accumulated on the membrane surface as fouling and/or a concentration polarization layer.

The final permeate fraction contained less than 10 mg/L protein, approximately 140 mg/L carbohydrate and 280 mg/L ash. The passage of monovalent salts and ions, and monosaccharides probably accounts for the greater proportion of these values. The mean rejection of total solids exhibited by this membrane was 86.3%. This rate of rejection may be suitable for processing fiber isolation effluents. However, this rate would likely be inadequate for the treatment of effluent sources containing high concentration of solutes (e.g. protein solution effluent).

Permeate flux values for this trial are shown in Table 4.13. Although the feedwaters used in the trial were

TABLE 4.13. Filmtec Nanofiltration Permeate Flux

Time (min)	Feedwater Temperature (^{o}C)	Permeate Flux ($L/m^2/hr$)
5	11	42.9
30	14	29.4
60	18	24.9

low in solutes, and free of turbidity, a considerable reduction in flux occurred over the short period of processing. Approximately 42% of the initial flux was lost before a 75% rate of permeate recovery was reached. This indicated that the fiber effluent possessed solutes capable of fouling this membrane very quickly.

4.4. Reverse Osmosis Studies

4.4.1. Feedwater Source

Two effluent sources were used in the RO trials conducted at Woodstone Foods. Again, the primary desludger effluent was chosen due to the high total solids content of this water. Secondly, process water from the plant's hull fiber isolation line was used as requested by Woodstone Foods. Although an increasingly large volume of this water was discharged from the plant, it was lower in total solids than other process streams.

The temperature of the protein desludger and fiber screen effluents used in these trials are included in Table 4.13. No standard conditions of pH and temperature was set for the fiber isolation process as the isolation step was a physical rather than chemical separation. The effluent from this process varied according to the temperature of incoming municipal water.

Collection of fiber screen effluent was accomplished by filling a 500 L feedwater tank directly from the process

discharge. The collection of the desludger effluents consisted of pumping the process discharge through a heat exchanger before entering the RO feedwater tank.

4.4.2. Evaluation of Membrane Rejection

Unlike UF, reverse osmosis membranes have the ability to retain ions, salts and other small molecular weight solutes. Sodium chloride is a standard salt normally used to evaluate solute retention properties of a membrane. As membrane production methods are not always consistent and flawless, the rejection (retention) efficiency of each RO membrane is normally checked.

The results of the initial rejection evaluation of Osmonics 192 HR and 192 SR modules is provided in Tables 4.14. and 4.15. Manufacturer specifications for these modules are 97.5% (96% minimum) and 95% (92.5% minimum) NaCl rejection, respectively (Osmonics, 1984). These data are calculated after 30 minutes of system operation. These tables indicate that rejection values of 96.9% and 93.6% were obtained under similar operating conditions. Thus, minimum rejection specifications were met during evaluation trials.

Similarly, the salt rejection value of Filmtec's TWS-2026 modules met the published specifications of 98% (96% minimum). The value obtained at 30 minutes was 96.9% (Table 4.16.).

TABLE 4.14. Determination of Salt Rejection - Osmo 192 HR

Time (min)	NaCl (mg/L)		Rejection (%)	Flow Rate (mL/min)		Recovery (%)
	Concentrate	Permeate		Concentrate	Permeate	
5	2800	<50	100.0	800	200	80.0
30	4400	100	96.9	760	200	79.2
60	5500	200	94.6	720	180	80.0
90	6100	300	92.6	700	180	79.6
120	7000	400	91.1	690	180	79.3
150	7400	400	91.5	680	165	80.5
180	7700	400	91.8	680	165	80.5
210	8000	400	92.0	670	150	81.7
240	8200	400	92.2	670	140	82.7
Mean			93.6			80.4

TABLE 4.15. Determination of Salt Rejection - Osmo 192 SR

Time (min)	NaCl (mg/L)		Rejection (%)	Flow Rate (mL/min)		Recovery (%)
	Concentrate	Permeate		Concentrate	Permeate	
5	2600	100	95.6	1030	250	83.7
30	4200	200	93.6	900	250	78.3
60	5000	300	91.4	880	250	77.8
90	5800	400	89.7	900	250	78.3
120	6500	500	88.2	900	200	81.8
150	6800	600	86.4	850	150	85.0
180	7200	600	87.0	840	150	84.9
210	7800	600	87.8	830	150	84.7
240	8000	600	88.0	820	150	84.5
Mean			89.7			82.1

TABLE 4.16. Determination of Salt Rejection - TW30-2026

Time (min)	NaCl (mg/L)		Rejection (%)	Flow Rate (mL/min)		Recovery (%)
	Concentrate	Permeate		Concentrate	Permeate	
5	1600	28	98.9	650	200	76.5
30	2380	52	96.9	570	170	77.0
60	2890	60	96.9	500	90	84.8
90	4930	206	93.1	430	90	82.7
120	5113	208	93.2	260	90	74.5
Mean			95.8			79.1

Average rejection values for a longer test period is also presented in Tables 4.14. to 4.16., providing data more representative of actual operating conditions. Means of 93.6, 89.7 and 95.8% were obtained during extended evaluation of the 192 HR, 192 SR and TW30-2026 membranes, respectively. Although these results are slightly lower than the manufacturer claims, the membranes are still acceptable for their intended use.

4.4.3. Reverse Osmosis Fraction Composition

4.4.3.1. Protein Effluent Fraction Analysis

Analysis data of the protein effluent RO fractions obtained using Osmonics' 192 HR, 192 SR and Filmtec's TW30-2026 modules are provided in Tables 4.17., 4.18. and 4.19., respectively. All effluent components tested for appeared to be well retained by the membranes. The components were concentrated proportionately to the reduction in fluid volume due to permeate passage. Carbohydrate accounted for approximately 75% of the total solids in the concentrates. Ash was the second largest component at 20% and protein was third at 5%. The ash contained 75 to 93% sodium chloride. This salt resulted from the use of NaOH and HCl for pH adjustment in the protein isolation step.

All three membranes tested produced a high quality permeate water (Tables 4.17. to 4.19.). Low levels of tannin (< 5 mg/l) and COD (< 200 mg/L) were found in the permeate

TABLE 4.17. Analysis of Permeate and Concentrate Fractions from RO of Protein Desludger Effluent (mg/L) - Osmo 192SR

Time (min)	COD	Total Solid	Carbohydrate[1]	Protein	Tannin	NaCl	Ash	pH
Feedwater Analysis								
0	12800	14700	11800	745	162	2300	2178	4.43
Concentrate Analysis								
5	18000	21200	17100	785	298	3340	3296	4.57
30	23600	29760	24700	1050	349	3890	3980	4.55
60	29700	33840	27700	1230	397	4630	4924	4.52
90	44400	50390	43800	1390	445	5150	5188	4.50
120	50000	55280	47800	1510	493	5850	5996	4.48
150	51700	58010	50200	1610	528	5910	6232	4.48
180	54900	59800	51800	1720	557	5970	6300	4.48
210	54200	60230	52100	1810	560	6090	6340	4.45
240	54700	61120	52900	1910	544	5910	6272	4.48
270	54400	61430	53200	1990	552	5910	6256	4.48
300	54700	62900	54500	2050	576	6150	6320	4.49
Permeate Analysis								
5	120	220	75	<1	<5	140	144	3.96
30	120	324	108	<1	<5	220	216	3.75
60	119	412	76	<1	<5	310	336	3.69
90	125	488	112	<1	<5	370	376	3.69
120	135	568	64	<1	<5	500	504	3.70
150	143	676	112	<1	<5	540	564	3.70
180	144	696	108	<1	<5	550	588	3.71
210	160	712	100	<1	<5	560	612	3.72
240	153	700	116	<1	<5	550	616	3.73
270	159	760	120	<1	<5	580	640	3.74
300	167	800	136	<1	<5	600	664	3.76

[1]Calculated by difference

TABLE 4.18. Analysis of Permeate and Concentrate Fractions from RO of Protein Desludger Effluent (mg/L) - Osmo 192HR

Time (min)	COD	Total Solids	Carbohydrate[1]	Protein	Tannin	NaCl	Ash	pH
Feedwater Analysis								
0	13900	17650	12800	656	191	3700	4220	4.32
Concentrate Analysis								
5	17000	20440	13800	325	280	5000	5825	4.58
30	31200	36120	25300	990	378	8200	9860	4.28
60	39000	48400	36190	1100	500	9500	11110	4.15
90	43500	55500	51600	1230	592	10500	12630	4.14
120	48000	57450	43400	1280	584	11000	12780	4.12
150	48800	59980	45700	1200	584	11300	13070	4.14
180	46500	62510	48100	1310	576	10000	13110	4.14
210	48800	64090	49200	1395	624	10300	13480	4.15
240	49730	66830	51600	1350	669	10600	13930	4.19
270	55600	69310	53500	1425	696	11000	14420	4.20
300	54880	74220	57800	1590	736	10900	14800	4.19
330	64600	76560	59800	1770	792	11200	14960	4.20
360	65300	78610	61600	1950	776	11300	15030	4.20
Permeate Analysis								
5	38	344	54	<2	<5	300	288	4.20
30	51	732	65	<2	<5	700	666	3.90
60	68	861	106	<2	<5	900	753	3.82
90	87	998	104	<2	<5	1000	892	3.80
120	102	983	77	<2	<5	1000	904	3.80
150	113	964	82	<2	<5	1000	880	3.82
180	123	1002	122	<2	<5	1000	878	3.82
210	132	1064	70	<2	<5	1000	994	3.84
240	138	1275	161	<2	<5	1100	1112	3.86
270	141	1332	150	<2	<5	1200	1180	3.88
300	147	1491	162	<2	<5	1300	1327	3.88
330	158	1468	76	<2	<5	1400	1390	3.88
360	165	1436	98	<2	<5	1400	1336	3.88

[1]Calculated by difference

TABLE 4.19. Analysis of Permeate and Concentrate Fractions from RO of Protein Desludger Effluent (mg/L) - TW30-2026

Time (min)	COD	Total Solids	Carbohydrate[1]	Protein	Tannin	NaCl	Ash	pH
Feedwater Analysis								
0	22840	26530	21360	581	296	4370	4692	4.38
Concentrate Analysis								
5	26910	31200	25210	399	460	5260	5668	4.43
60	44970	53980	43810	767	850	8910	10170	4.39
120	55210	66890	53150	1020	1040	11050	12720	4.39
180	65600	76540	61320	1330	1150	11960	13890	4.38
240	69190	82710	66590	1510	1280	12620	14610	4.38
300	72000	86200	69070	1660	1280	13530	15470	4.39
360	74730	88690	71840	1740	1280	13280	15110	4.38
Permeate Analysis								
5	<10	164	11	<1	<5	124	152	4.91
60	<10	388	19	<1	<5	302	368	4.99
120	<10	584	15	<1	<5	483	568	4.99
180	<10	646	25	<1	<5	590	620	5.03
240	19	692	33	<1	<5	607	658	4.99
300	45	694	41	<1	<5	644	652	5.03
360	42	676	37	<1	<5	628	638	5.03

[1]Calculated by difference

water. These permeate characteristics are desirable to prevent the transfer of flavour and colour to food products, if this water is recycled to plant processes on scale up. The passage of ash was considerably higher than the passage of organic components to the permeate. This was expected since the membranes retain the higher molecular weight organic components more effectively than the low molecular weight ions, salts, etc.. However, the presence of salts and other inorganic compounds, at these levels, may act to improve the solubilization and resulting extraction of a protein isolate on water recyclization.

4.4.3.2. Fiber Effluent Fraction Analysis

Tables 4.20., 4.21. and 4.22. show the analysis results of the fiber effluent 20 fractions using Osmonics' 192 HR, 192 SR and Filmtec's TW30-2026 modules, respectively. In these trials, the organic constituents measured as COD, carbohydrate, tannin and protein were only concentrated 2-3X, while the inorganic (ash) component was concentrated 4X. A 4X concentration of organic material was expected as the RO system was operated at a 77.4% recovery rate. However, all three modules exhibited effective rejection of organic molecules, shown by the low levels present in the permeate water. This "missing" organic material may be accounted for in a layer of fouling likely present on the membrane surface. The small loss of organic material may not have been noticed in previous protein effluent trials due to the

TABLE 4.20. Analysis of Permeate and Concentrate Fractions from RO of Fiber Isolation Effluents (mg/L) - Osmo 192HR

Time	COD	Total Solids	Carbohydrate[1]	Protein	Tannin	NaCl	Ash	pH
Feedwater Analysis								
0	676	1200	681	83	32	400	436	4.50
Concentrate Analysis								
5	726	1496	722	98	35	700	676	5.35
30	826	1740	787	105	45	900	848	5.32
60	1050	2148	1004	120	45	1100	1024	5.30
90	1230	2596	1266	120	50	1200	1210	5.30
120	1350	2924	1399	120	56	1300	1405	5.30
150	1500	3192	1510	135	56	1400	1524	5.30
180	1650	3204	1522	135	64	1500	1547	5.32
210	1710	3350	1614	150	66	1500	1587	5.32
240	1700	3492	1787	165	64	1400	1540	5.32
270	1680	3560	1767	173	67	1500	1620	5.32
300	1700	3584	1772	180	67	1500	1632	5.32
Permeate Analysis								
5	<15	120	67	<1	<5	<100	52	5.15
30	<15	108	55	<1	<5	<100	52	5.20
60	<15	114	53	<1	<5	<100	60	5.30
90	<15	118	37	<1	<5	<100	80	5.30
120	<15	138	41	<1	<5	<100	96	5.20
150	<15	152	30	<1	<5	<100	122	5.40
180	<15	180	66	<1	<5	<100	114	5.35
210	<15	168	31	<1	<5	<100	136	5.30
240	<15	170	44	<1	<5	<100	125	5.35
270	<15	174	46	<1	<5	<100	127	5.30
300	<15	176	43	<1	<5	<100	132	5.30

[1]Calculated by difference

TABLE 4.21. Analysis of Permeate and Concentrate Fractions from RO of Fiber Isolation Effluent (mg/L) - Osmo 192SR

Time	COD	Total Solids	Carbohydrate[1]	Protein	Tannin	NaCl	Ash	pH
Feedwater Analysis								
0	2400	2723	1990	308	44	400	422	6.45
Concentrate Analysis								
5	2760	2947	1970	297	50	600	683	6.40
30	3600	4120	2420	502	68	1100	1202	6.52
60	4510	4833	2790	620	81	1300	1427	6.57
90	5560	5780	3410	731	96	1500	1638	6.67
120	5410	6285	3760	818	102	1500	1711	6.71
150	5260	7275	4670	834	108	1500	1774	6.64
180	6760	7667	5090	755	114	1600	1825	6.61
210	6010	7905	5300	762	116	1600	1846	6.64
240	6010	7790	5130	794	122	1600	1863	6.66
270	7510	8120	5400	849	132	1700	1876	6.69
300	7210	8011	5200	912	136	1700	1895	6.71
330	7510	8175	5260	912	146	1700	1921	6.73
360	7510	8175	5360	905	148	1700	1907	6.75
Permeate Analysis								
5	53	69	48	<1	<5	<100	20	5.30
30	45	64	38	<1	<5	<100	25	5.15
60	42	57	20	<1	<5	<100	38	5.15
90	38	61	18	<1	<5	<100	42	5.23
120	38	66	15	<1	<5	<100	50	5.30
150	36	47	20	<1	<5	<100	53	5.32
180	38	80	20	<1	<5	<100	59	5.44
210	42	89	36	<1	<5	<100	52	5.47
240	36	95	34	<1	<5	<100	60	5.54
270	38	92	21	<1	<5	<100	70	5.51
300	38	101	28	<1	<5	<100	72	5.58
330	36	105	29	<1	<5	<100	75	5.56
360	38	102	30	<1	<5	<100	71	5.55

[1]Calculated by difference

TABLE 4.22. Analysis of Permeate and Concentrate Fractions from RO of Fiber Isolation Effluent (mg/L) - TW30-2026

Time (min)	COD	Total Solids	Carbohydrate[1]	Protein	Tannin	NaCl	Ash	pH
Feedwater Analysis								
0	2470	2828	1760	565	63	387	508	6.04
Concentrate Analysis								
5	1350	2140	826	298	65	780	1016	5.75
60	3280	4764	2500	733	134	1300	1536	5.92
120	4090	5462	2790	953	165	1510	1724	6.37
180	4570	5960	3070	1030	182	1600	1856	6.50
240	4720	6436	3360	1160	189	1660	1921	6.64
300	4910	6760	3780	1020	191	1690	1964	6.67
360	5030	7045	4010	1040	191	1720	2000	6.71
Permeate Analysis								
5	<10	42	11	<1	<1	18	31	4.93
60	<10	63	18	<1	<1	25	45	5.06
120	<10	84	33	<1	<1	26	51	5.56
180	<10	90	36	<1	<1	22	54	5.80
240	<10	93	35	<1	<1	24	58	5.83
300	<10	92	32	<1	<1	28	60	5.95
360	15	92	33	<1	<1	30	59	5.94

[1]Calculated by difference

high concentration of solids present in these waters.

The feed effluents ranged considerably in concentration and composition (Tables 4.20. to 4.22.). Total solids in the feedwater ranged from 1200 to 2828 mg/L. This effluent source was relatively low in total solids compared to the primary and secondary protein desludgers. Suspended solids comprise 30-50% of the total solids and should be removed by a high capacity filtration system. The resulting filtrate may be recycled back into plant processes without further treatment.

Carbohydrate in the concentrate and feedwater ranged from 50-65% of the total solids. Ash in these waters ranged from 20-50%, while the protein content ranges from 5-20%. As all fiber effluent samples contained less than 900 mg/L protein and up to 1700 mg/L carbohydrate, the recovery of a protein fraction would likely not be very practical.

The permeate water produced by all three membranes was low in total solids (< 200 mg/L) and could be discharged into the municipal sewer system without penalty. However, the reuse of this water in preceeding processes would prove to be the most resource conscious and economical alternative.

4.4.3.3. Qualitative Analysis

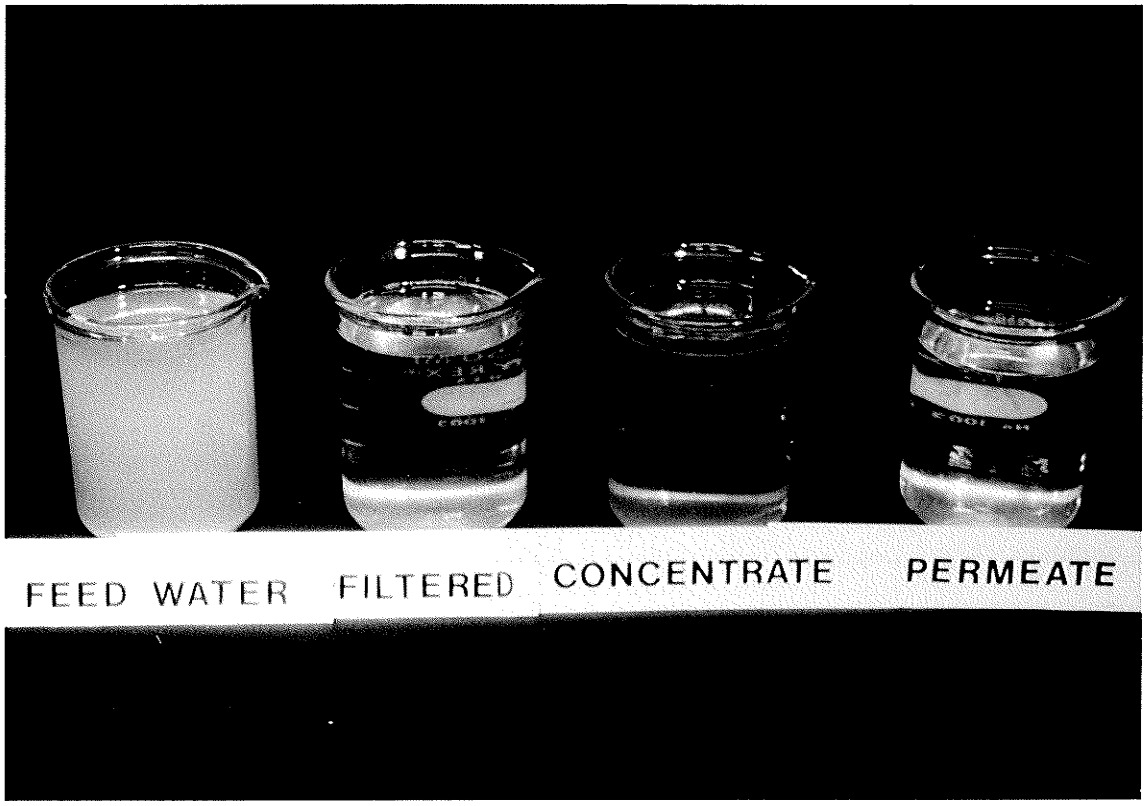
The concentrate fraction produced by the protein effluent feedwaters were grey-green in color and metallic

pea-like in aroma. This fraction also appeared to be slightly viscous and turbid. Corrosion of the brass pre-filter canisters on the Ajax 500 unit was likely responsible for the color phenomenon. In commercial applications, these, and related cuprous parts would be replaced by food quality plastic or stainless steel parts. It was also noted that upon storage at room temperature (22^{o}C), the concentrates produced a large amount of coarse, white sediment. The more concentrated fractions produced larger amounts of sediment. The nature of the sediment was not analyzed but may have been due to protein precipitation from increased salt concentrations or a carbohydrate-rich substance formed during cooling of the concentrated solution.

The concentrate obtained from the fiber effluent trials was similar in odor and turbidity but lacked the characteristic green color of the protein effluent trials. The relatively low levels of ash and particularly NaCl in the concentrate likely prevented the color complex from forming. The resulting fractions from the reverse osmosis operation are shown in Fig. 4.6.

Permeate fractions resulting from all RO trials were free of color and turbidity. A slight off-pea odor could be detected in these water. This source of "restored" water was of high quality and would be suitable for reuse in plant processes.

Figure 4.6. Fractions from the RO of fiber effluent.


FEED WATER
FILTERED
CONCENTRATE
PERMEATE

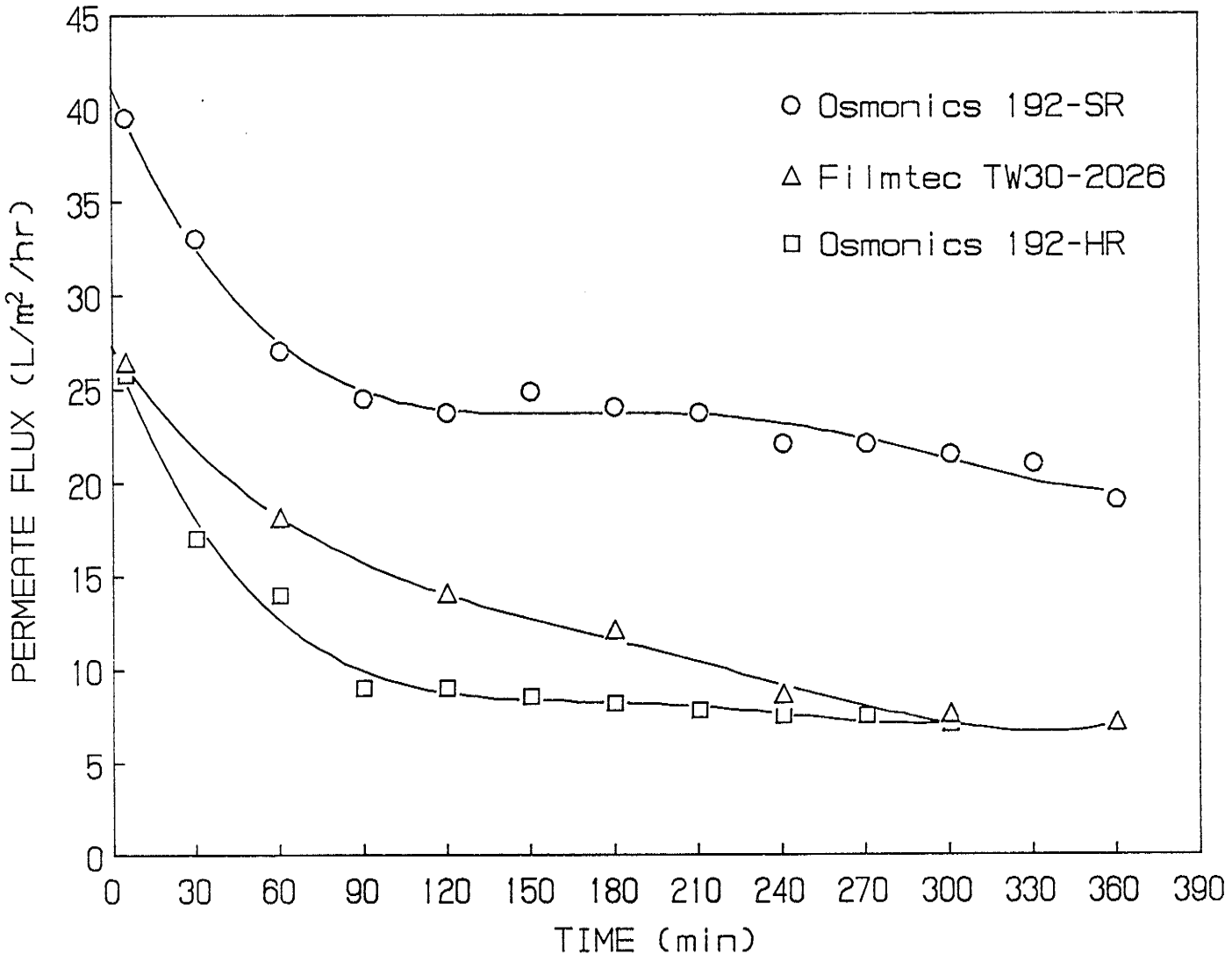
4.4.4. Reverse Osmosis Permeate Flux

The decline of permeate flux during the membrane treatment of Woodstone's protein desludger effluent is shown in Fig. 4.7. These trials consisted of processing the effluent, in a continuous mode, for a period of six hours. This time length was required by Woodstone Foods as it was the minimum period that the RO unit could be economically operated before cleaning was required. The trial using Osmonics 197-HR module was terminated one hour early due to problems with the regulation of recovery at low flux rates.

During these trials, mean permeate values of 24.7, 13.2, and 11.0 L/m^2hr were recorded for the 192 SR , TW30-2026 and 192 HR modules, respectively. The mean reduction in permeate flux over the period of processing was 53% for the 192 SR module, 73% for the TW30-2026 module and 74% for the 192 HR module. Thus, the 192 HR module provided approximately twice the permeate flow afforded by the other two units tested. Also, the reduction in permeate flow due to membrane fouling and/or concentration polarization was considerably less using this module. However, it should be noted that the operating pressure used on the Filmtec TW30-2026 module was only 21.1 Kg/cm^2 compared to 35.2 Kg/cm^2 used with the Osmonics modules. The lower pressure was used in order to comply with manufacturer's recommendations for this module.

Filmtec Inc. also manufactures larger 10 cm and 20 cm

Figure 4.7. Effect of time on RO permeate flux using protein desludger effluent feedwater.


45
40
35
30
25
20
15
10
5
0
0
30
60
90
120
150
180
210
240
270
300
330
360
390
PERMEATE FLUX (L/m²/hr)
TIME (min)
Osmonics 192-SR
Filmtec TW30-2026
Osmonics 192-HR

diameter modules suitable for industrial applications. These larger modules can withstand operating pressures up to 42.2 kg/cm^2. The higher pressures would induce an increased production of permeate per area of membrane. In addition, higher pressures may be beneficial in improving membrane performance adversely affected by the increased osmotic pressures during concentration.

The change in permeate flux, during membrane treatment of Woodstone's fiber isolation effluent, is shown in Fig. 4.8. During these trials, mean permeate flux values of 25.9, 16.6 and 12.2 L/m^2hr were recorded for the 192 SR, 192 HR and TW30-2026 modules, respectively. The overall drop in flux during processing was 67% for the 192 SR module, 68% for the 192 SR module and 75% for the TW30-2026 module. Again, as with the protein effluent, the 192 SR module appeared to possess superior performance characteristics. The Filmtec module again encountered the largest loss in flux during effluent processing. This result was not anticipated, as the feed waters were prefiltered, for the removal of suspended solids, using the five micron cartridges. Blinding of the prefilter cartridges, by feedwater effluents, is shown in Fig. 4.9. Increased flux losses may be a phenomenon associated with the use of a lower operating pressure during processing.

4.4.5. Effect of Membrane Cleaning on Permeate Flux

After the RO trials were completed, the modules were

Figure 4.8. Effect of time on RO permeate flux using fiber decanter effluent feedwater.

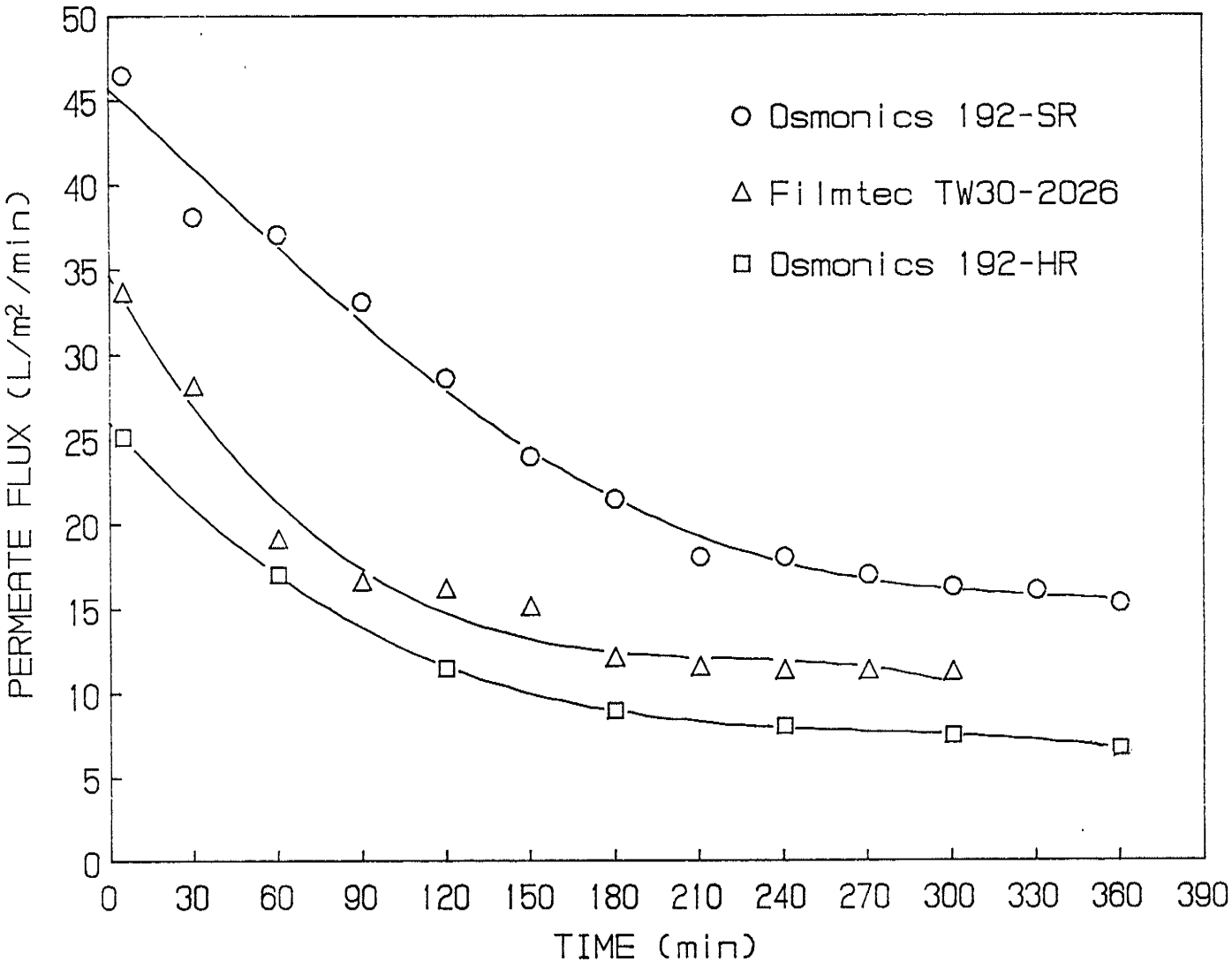
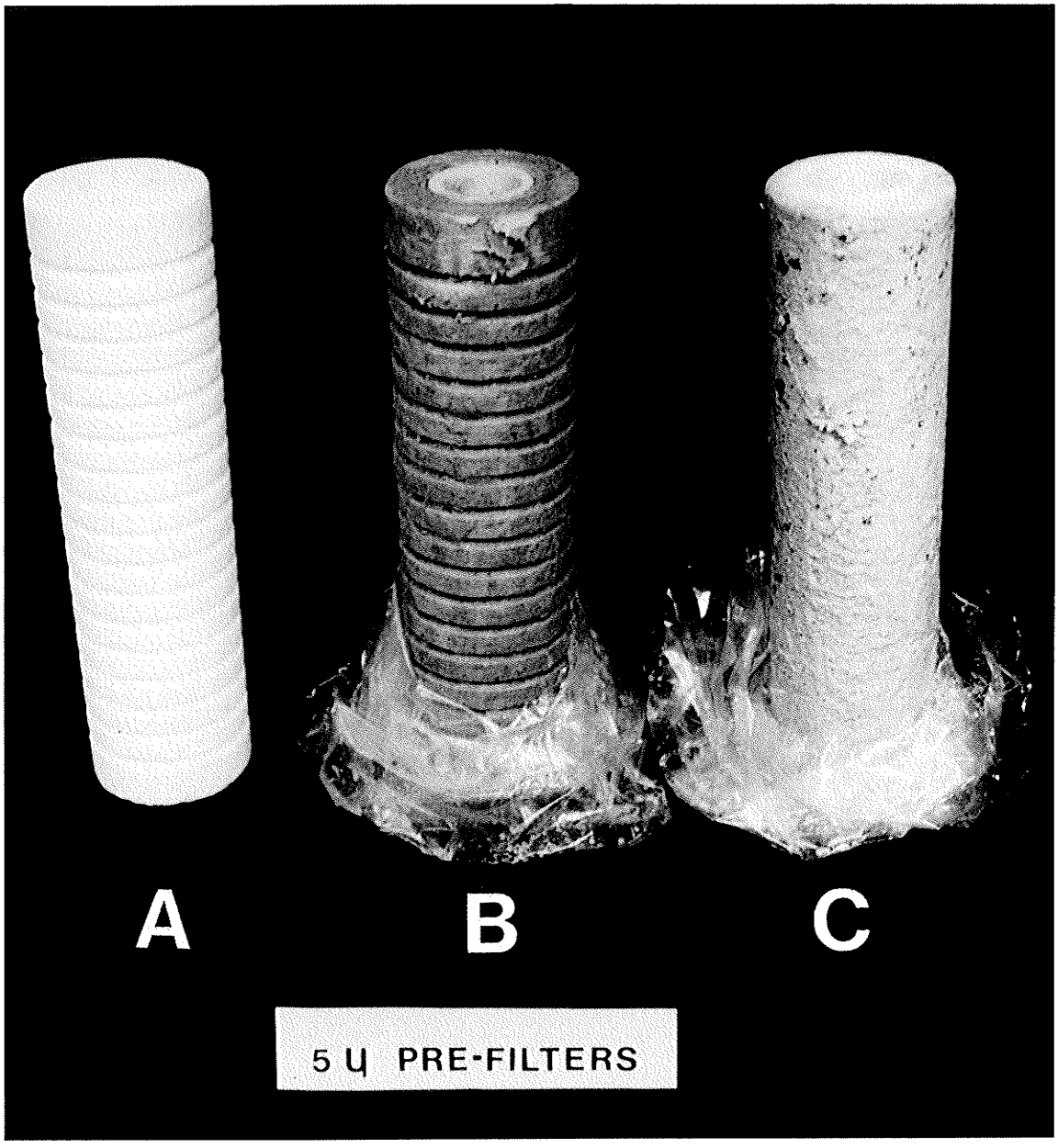

PERMEATE FLUX (L/m²/min)
TIME (min)
Osmonics 192-SR
Filmtec TW30-2026
Osmonics 192-HR
0
5
10
15
20
25
30
35
40
45
50
0
30
60
90
120
150
180
210
240
270
300
330
360
390

Figure 4.9. Blinded 5μ prefilter cartridges.

A. New prefilter cartridge.

B. Protein desludger effluent blinded cartridge.

C. Fiber isolation effluent blinded cartridge.

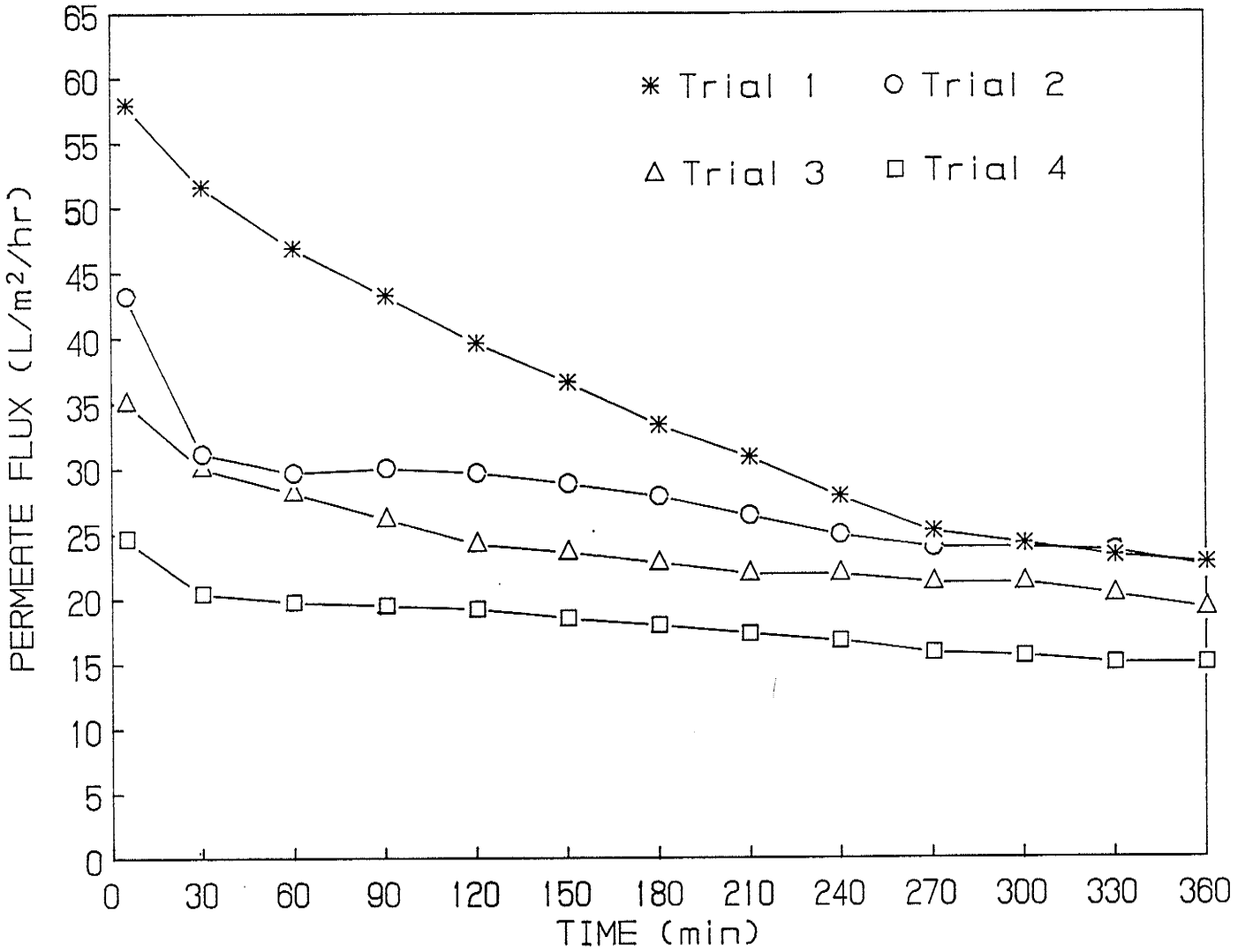

A
B
C
5 µ PRE-FILTERS

flushed with permeate water and cleaned. Permeate water is often chosen in commercial practices for flushing and cleaning membranes, as it is a source of purified water possessing a suitable pH and temperature.

The results of a study undertaken to monitor the effect of manufacturer recommended cleaning procedures, on membrane flux, is illustrated in Fig. 4.10. In this experiment the Osmonics' 192 SR module was subjected to a series of four, six hour runs using effluent from the hull fiber isolation process. The initial permeate flux using the plant's fresh water source was 48.5 $L/m^2/hr$. After the four effluent processing runs and cleaning cycles were completed, the flux of permeate was measured at less than 18.5 $L/m^2/hr$ using fresh plant water as feed. This 62% loss in permeate flux was not permanent. The membranes were allowed to sit for two days in a 0.2% formaldehyde storage solution. After this period, the flux was measured again and then the membranes were recleaned. The flux had increased to 40 $L/m^2/hr$ after storage, and been restored to 48 $L/m^2/hr$ after recleaning. This "recovery" of flux on storage may be due to a loosening of the compacted fouling layer over time. The loosened layer could be then subject to removal by turbulent recleaning (Applegate, 1984).

The permeate flux did not decrease proportionately throughout the runs (Fig. 4.6.). This indicated that the incomplete cleaning action afforded by the manufacturer supplied cleaning compounds (Osmonics Ultrazyme 73 and NP-

Figure 4.10. Effect of incomplete cleaning on RO permeate flux over time.


* Trial 1
o Trial 2
Δ Trial 3
□ Trial 4
PERMEATE FLUX (L/m²/hr)
TIME (min)
0
5
10
15
20
25
30
35
40
45
50
55
60
65
0
30
60
90
120
150
180
210
240
270
300
330
360

23) was more evident at the start of a run than at the end. Overall, these compounds were ineffective in restoring flux losses due to membrane fouling.

Additional attempts were made at cleaning the Osmonics membranes fouled by processing fiber effluents. The first compound used was Bird Archer's acid-based Formulae 2195 liquid resin cleaner. This cleaner was supplied as a liquid which produced a pH of 2.5 on dilution. Osmonics' powdered Ultrazyme 93 enzyme cleaner and NP 20 liquid surfactant were used in combination during a second membrane cleaning evaluation. Each cleaning trial was run for an hour according to manufacturer's recommendations. Both cleaning solutions were ineffective as less than 25% of the original permeate flux was restored. However, the original flux was renewed after the RO modules were stored in a 0.2% formaldehyde solution for several days and then recleaned.

Further cleaning trials were undertaken on Osmonic modules fouled by protein effluent. The two solutions used in these trials were a Lactonase enzyme cleaner and a 0.1% NaOH/0.5% sodium dodecylsulfate solution. The Lactonase cleaner restored 70% of the flux while the NaOH/SDS solution restored less than 25% of the flux lost from fouling. As before, the original flux was restored by storage in a formaldehyde solution and recleaning with Lactonase or Ultrazyme 73 cleaner.

Later cleaning trials were undertaken using Zenon's MC-78 membrane cleaner on the Filmtec TW30-2026 RO module. One hour cleaning cycles were performed according to the manufacturer's recommendations. This cleaner was successful in restoring 100% of the flux lost from protein effluent fouling and approximately 75% of the flux lost from fiber effluent fouling. The MC 78 cleaner appeared to be effective on Filmtec's thin film composite membrane but has not been tested with cellulose acetate modules.

4.5. Laboratory Simulated Effluent Recycle Studies

4.5.1. Primary Effluent Recycle

This study was undertaken to evaluate the effect of recycling primary and secondary effluent on the compositional quality of the derived protein isolate and the resulting effluent. Table 4.23. and Fig. 4.11. demonstrate the increase of components found in primary protein isolation effluents when reused in the protein isolation process. The soluble components did not increase proportionately as the cycles increased. This indicated that the solubilization of components from the pea material was decreasing and should cause an increase in protein isolate yield.

The analysis results of the protein isolates obtained on reuse of the primary effluents in the process are shown in Fig. 4.12. and Table 4.24. The protein content decreased

Table 4.23. Pea Effluent Recycle Composition - Step 1

Sample-Cycle	COD (mg/L)	Total Solids (mg/L)	Carbohydrate (mg/L)	Protein (mg/L)	Tannin (mg/L)	Ash (mg/L)
1'D-1	22400	29900	28600	3520	280	7730
1'D-2[2]	37200	49200	28100	5810	482	12330
1'D-3	47300	61500	38900	8020	514	14580
2'D-1	6910	9600	6220	733	100	2631
2'D-2	10700	13900	8980	1410	142	3506
2'D-3	14600	18200	12400	1880	181	3946

[2]1'D-2 = Primary desludger effluent from the second cycle of protein isolation

Table 4.24. Protein Isolate Composition (dwb) - Step 1

Cycle	Protein (%)	Carbohydrate[1] (%)	Ash (%)
1	88.9	7.5	3.6
2	86.7	9.5	3.8
3	86.0	9.8	4.2

[1]Calculated by difference

Figure 4.11. Composition of protein isolation effluent on recycle - Step 1.

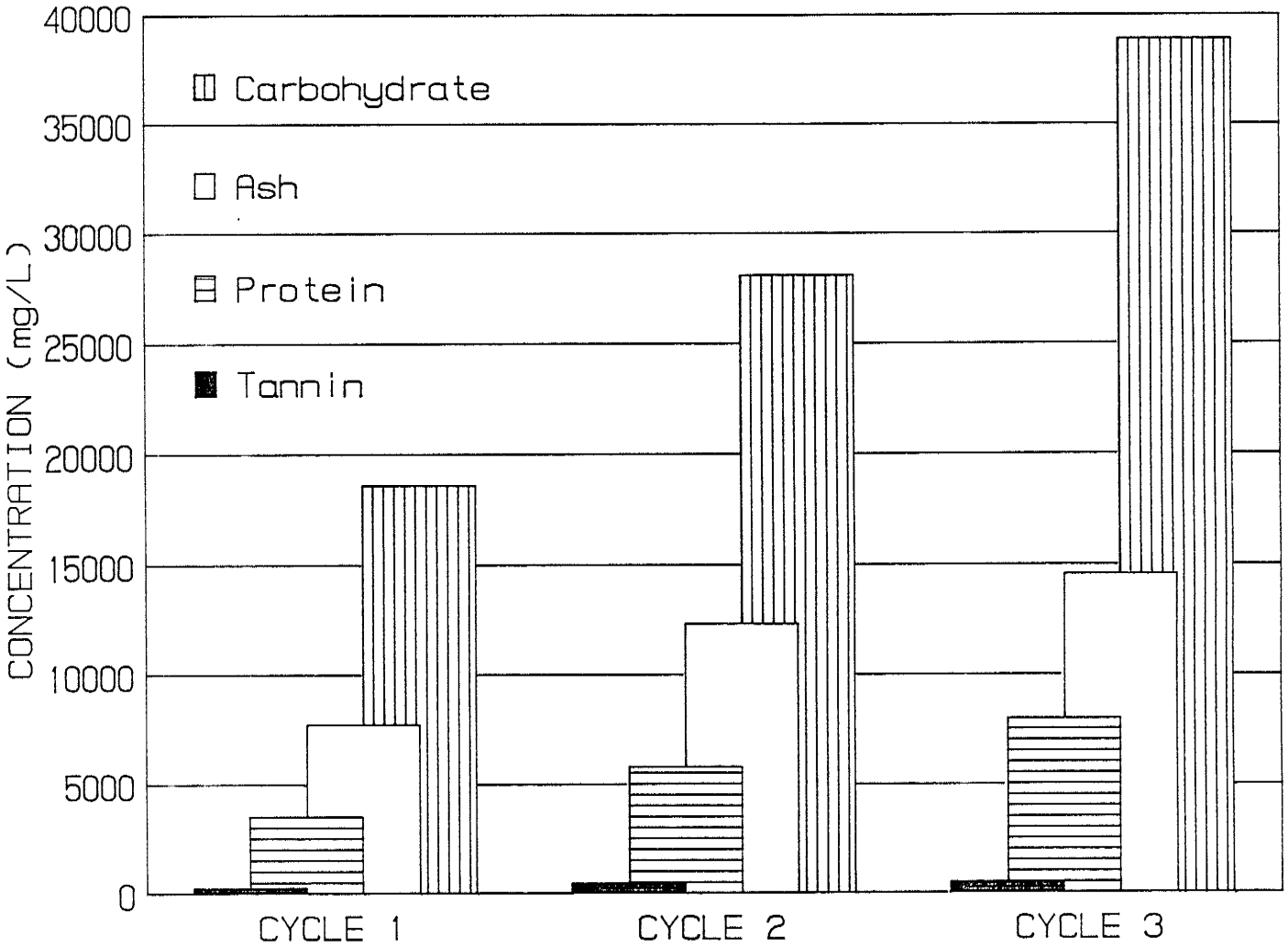
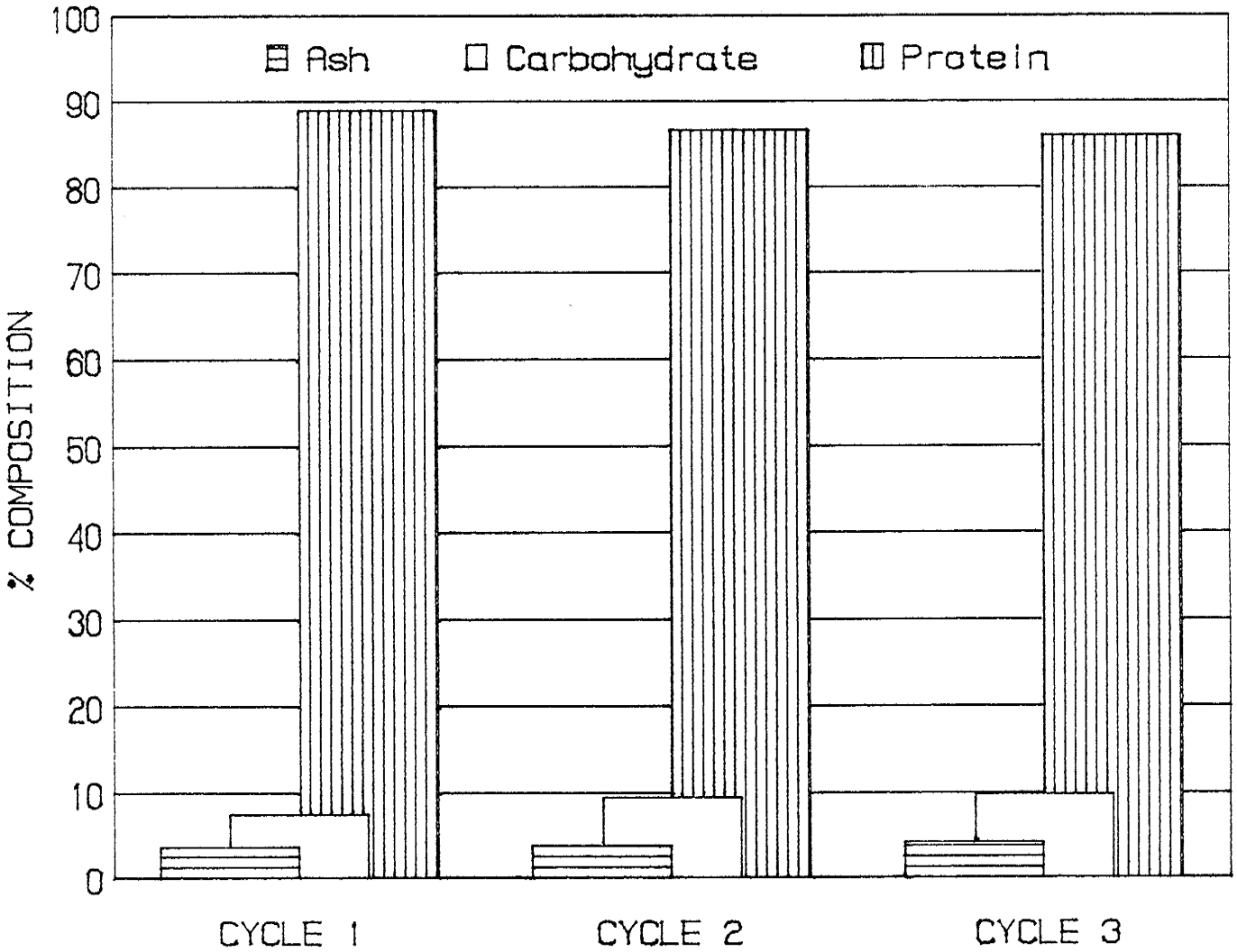

40000
35000
30000
25000
20000
15000
10000
5000
0
CONCENTRATION (mg/L)
Carbohydrate
Ash
Protein
Tannin
CYCLE 1
CYCLE 2
CYCLE 3

Figure 4.12. Composition of protein isolate on recycle of process waters - Step 1.


Ash
Carbohydrate
Protein
% COMPOSITION
100
90
80
70
60
50
40
30
20
10
0
CYCLE 1
CYCLE 2
CYCLE 3

slightly from 88.9 to 86% over the three cycles. Ash and carbohydrate increased slightly from 3.6 to 4.2% and 7.5 to 9.8%, respectively. Thus, the laboratory scale experiments showed a slight decrease in isolate purity possibly tolerable at the commercial level. If a recycling system is adopted by Woodstone Foods, protein functionality tests may be required to assess the physiochemical and functional attributes of these products.

4.5.2. Secondary Effluent Recycle

The results of Step 2 of the recycle experiment are provided in Tables 4.25, 4.26 and Figs. 4.13. and 4.14. The secondary effluents (protein wash) from the above trials were combined and used in a protein isolation cycle to evaluate the effect of using these waters.

The difference between the primary effluent collected from an isolation using fresh water (control) and an isolation using recycled secondary effluent is illustrated in Fig. 4.13. Cycle 1 of Step 1 was used as a control cycle. The protein isolates obtained from these two operations are compared in Fig. 4.14. The expected increase in solutes, present in the primary effluent, occurred. However, an unexpected increase in protein was found in the isolate (91.2%) as compared with the freshwater control isolate (88.9%). In addition, the carbohydrate and ash content of the isolate was found to decrease slightly when the recycled water was used. These results should be

TABLE 4.25. Pea Effluent Recycle Composition - Step 2

Sample	COD (mg/L)	Total Solid (mg/L)	Carbohydrate[2] (mg/L)	Protein (mg/L)	Tannin (mg/L)	Ash (mg/L)
Control[1]	8860	11040	6440	1330	115	3275
1' Eff.	31400	41900	26900	4430	502	10630
2' Eff.	8560	11050	7110	1130	116	2812
1'Control	22400	29900	28600	3520	280	7730
2'Control	6910	9600	6220	733	100	2631

[1]Starting Effluent for Recycle Step 2 (combined 2' effluent)
[2]Calculated by difference

TABLE 4.26. Protein Isolate Composition - Step 2

Sample	Protein (%)	Carbohydrate (%)	Ash (%)
Control	88.9	7.5	3.6
Recycle Isolate	91.2	5.5	3.3

Figure 4.13. Composition of protein isolation effluent on recycle - Step 2.

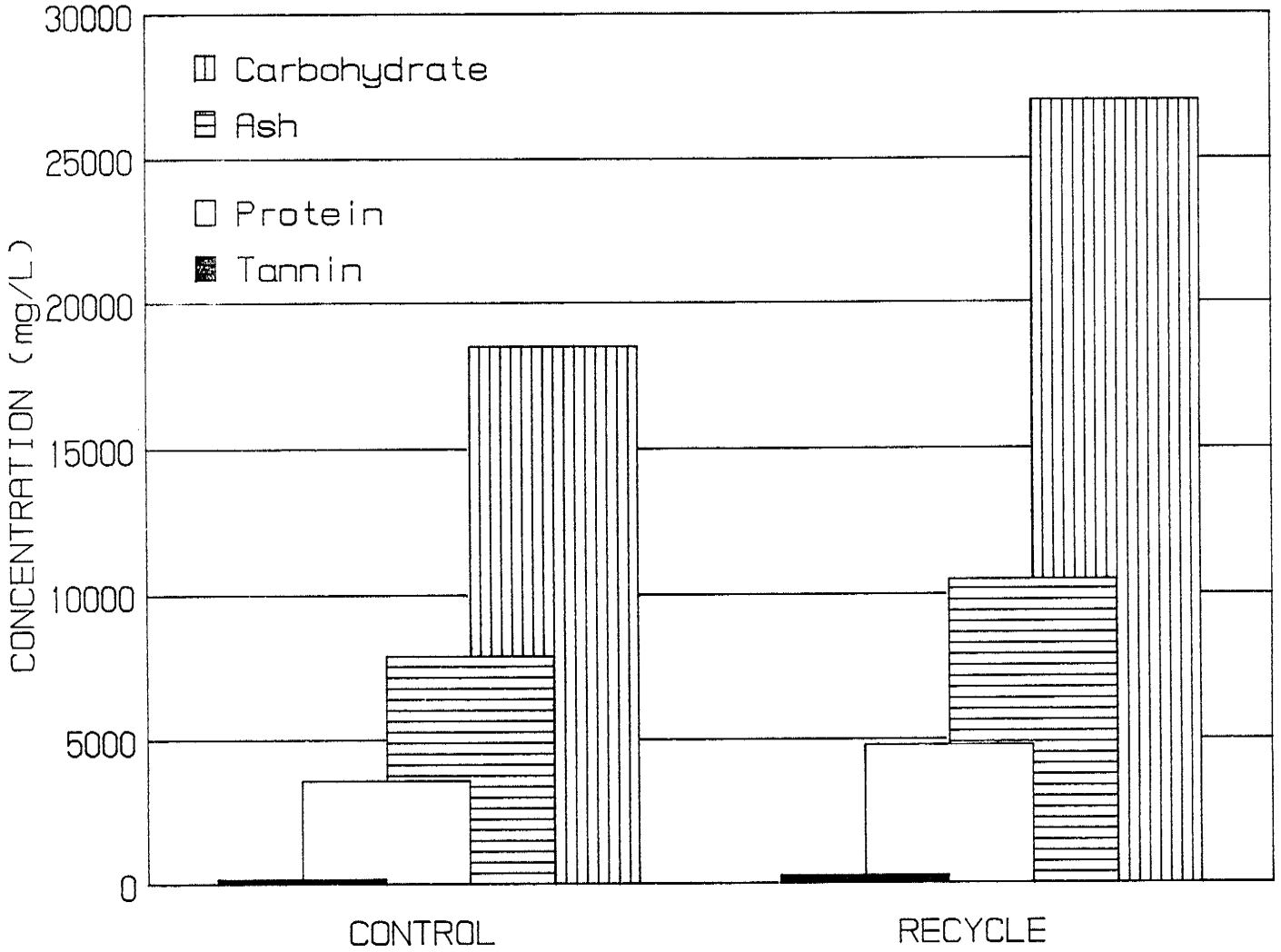
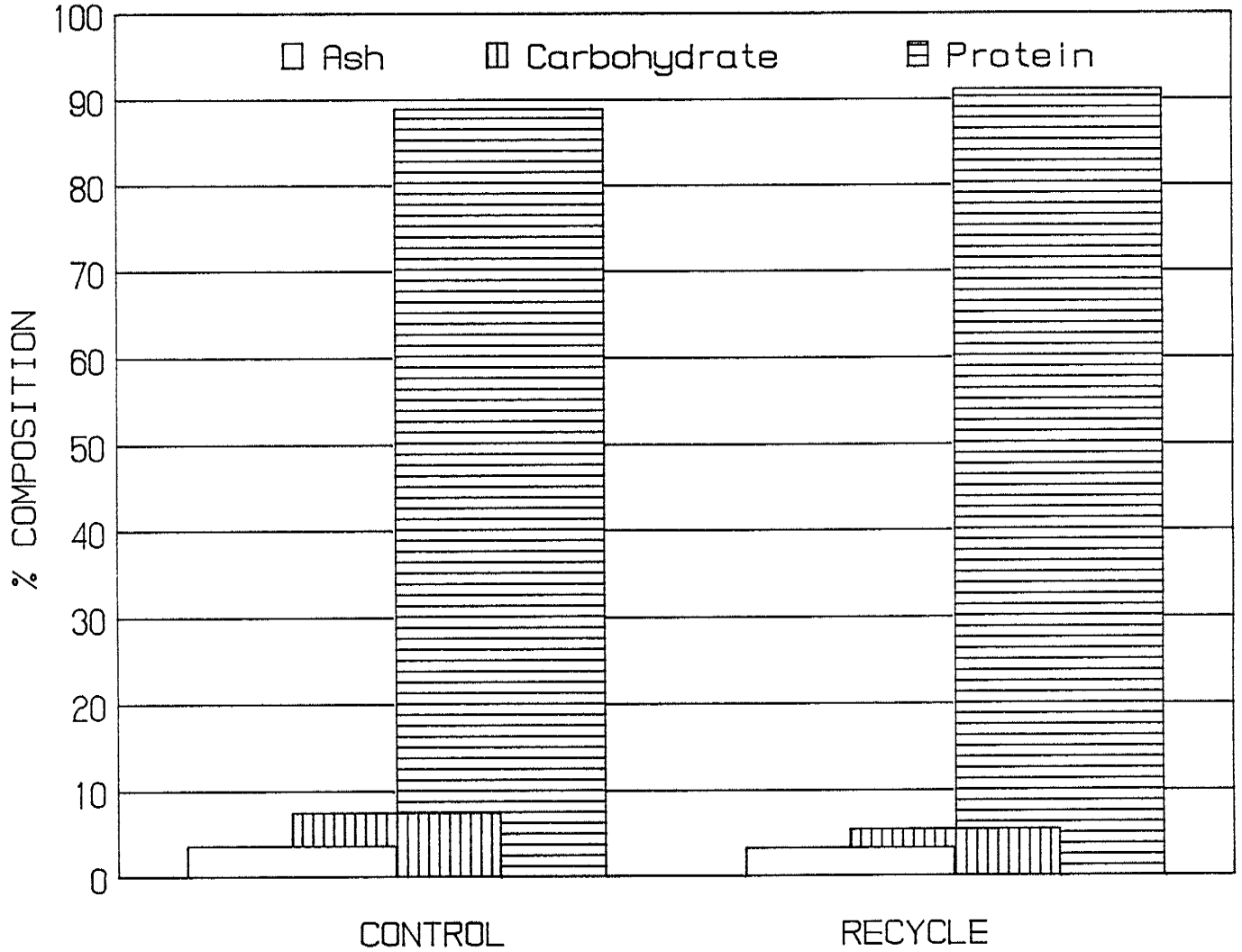

30000
25000
20000
15000
10000
5000
0
CONCENTRATION (mg/L)
Carbohydrate
Ash
Protein
Tannin
CONTROL
RECYCLE

Figure 4.14. Composition of protein isolate on recycle of process waters - Step 2.


Ash
Carbohydrate
Protein
% COMPOSITION
100
90
80
70
60
50
40
30
20
10
0
CONTROL
RECYCLE

checked on a pilot or plant scale experimental run to confirm the improvement of isolate purity when secondary effluents are recycled into the process. Visual inspection of the four protein isolates obtained in Steps 1 and 2 showed no evidence of color or odor change.

4.6. Effluent Floc Formation Studies

Due to difficulties encountered during previous in-house treatment attempts, concern was expressed by Woodstone Foods about the formation of a membrane fouling floc or precipitate in their effluent. This resulted in the undertaking of these studies. It was observed that a floc-like material formed in the protein isolation effluents upon cooling and/or membrane processing (Nickel, 1984). Studies were performed to evaluate the effect of temperature treatment on the formation of flocs in the protein effluents. In addition, $FeCl_3$ and a cellulase enzyme were evaluated for their ability to induce a floc in these effluents.

4.6.1. Floc Formation at Varied Temperatures

The results of the first temperature induced flocculation study are presented in Table 4.27. These data show that an increased amount of floc was formed as the treatment temperature increased. Approximately 3X as much floc was formed at 80-90^{o}C than at 4-45^{o}C. This formation of floc may be due to the denaturation of globular proteins

TABLE 4.27. Influence of Temperature on Floc Formation

Temperature (°C)	Time (hr)	Floc (mg/L)	Protein (%)	Carbohydrate[1] (%)	Ash (%)
90	1	406	76.6	19.3	4.1
80	1	404	77.4	18.8	3.8
70	1	332	77.2	20.3	2.5
60	1	336	--(1)	--	-
45	1	135	--	--	-
35	1	131	--	--	-
4	1	<10	--	--	-
4	24	137	--	--	-

[1]Calculated by difference
[2]-- = Not analysed due to small quantity of sample

TABLE 4.28. Influence of $FeCl_3$ on Floc Formation

2M $FeCl_3$ Concentration (mL/L)	Floc Formed (mg/L)	Observation[1] (at t = 1 hr) Turbidity	Sediment
0.0	24	-	-
0.2	336	+	-
0.5	611	+++	+
1.0	1081	++	+++
2.0	633	+++	+
3.0	284	+++	+
5.0	202	+++	-
10.0	197	+	-

[1] none= -, slight= +, moderate= ++, heavy= +++

present in the effluent. Decreased levels of this floc may form at sub-denaturation temperatures due to chemical instability of the protein structure at pH 4.5. This was supported by the observation that more than 75% of the floc was protein in nature.

4.6.2. Floc Formation with $FeCl_3$ Treatment

The data obtained during a study of the addition of $FeCl_3$ to protein isolation effluents is presented in Table 4.28. Approximately 1080 mg/L of floc was obtained upon the addition of 1 mL of 2M $FeCl_3$ per liter of effluent. A reduction in the amount of floc formed was noted when larger or smaller amounts of $FeCl_3$ was added. Visual inspection of these mixtures showed a heavy formation of floc. Although the amount of floc appeared to be considerable, further gravimetric analysis proved that only 11% of the organic material present in the test effluent was removed by the optimum amount of $FeCl_3$. However, it is not known if this treatment could remove as much floc, as through heating.

4.7. Antifoam Treatment of UF Feedwater

Foaming of the protein isolation effluent during ultrafiltration was found to cause operational difficulties. In some food processes, excess foaming occurs due to turbulent handling of process waters containing soluble proteins and/or the glycosides called saponins (McWatters and Cherry, 1977). In an industrial setting this problem

can be substantially important, as foaming can cause a loss of processing fluids as well as safety and sanitary considerations.

The data on the treatment of Woodstone's protein effluent with powdered and granular activated carbon is provided in Table 4.29. A range of dosages from 0.0 to 0.5 g/100 mL activated carbon was tested for foam capacity reduction. Both carbons tested were ineffective in reducing the foam capacity in protein effluents at the dosages used.

In contrast, Dow Corning's FG10 Antifoam Emulsion was effective in foam capacity reduction (Table 4.30.). At a 0.01% dose level the emulsion reduced the foam capacity of the effluent by about 87%. The foam capacity was completely inhibited at a 0.05% application level. However, the maximum level permissible in food products is 0.01% (FDA, 1986), and the addition of antifoam is costly. Therefore, emphasis should be placed on avoiding turbulent mixing and air incorporation, whenever possible.

TABLE 4.30. Influence of Activated Carbon on Foam Capacity

Treatment	Dosage (g/100 mL)				
	0.0	0.01	0.05	0.10	0.50
PAC	150 mL	150 mL	150 mL	150 mL	130 mL
GAC[1]	150 mL	150 mL	150 mL	150 mL	140 mL

[1]Granular Activated Carbon

TABLE 4.31. Influence of Antifoam on Foam Capacity

Treatment	Dosage (g/100 mL)				
	0.0	0.01	0.05	0.10	0.50
Foam (mL)	150	20	0	0	0

[1]Dow Corning FG-10 Antifoam Emulsion

5. CONCLUSIONS

In this study, field pea fractionation waters were characterized and subjected to ultrafiltration and reverse osmosis purification. Membrane performances and component rejections were evaluated as a means of recovering food by-products and reducing surcharges levied by local sewage treatment authorities. Work was undertaken at the laboratory scale to determine the effect of recycling process waters into the protein isolation operation.

Random sample analysis of Woodstones' process waters indicate a wide range of component concentrations. The total solids ranged from 830 mg/L for the fiber decanter to approximately 19000 mg/L for the primary protein desludgers with carbohydrate representing up to 50% of the organic loading. The variation in component concentrations indicates that the process waters should be handled as individual sources and not combined into a composite tank for treatment. Waters from the fiber and starch decanters and the pea wash could be recycled back into these processes as the water is used only as a carrier of particulate matter and is not needed for more critical solvent extractions. A 10-25 micron in-line backflushing filter could be useful in reducing the undesirable suspended solids prior to reuse.

Samples taken during the protein desludger sampling study were found to contain similar concentrations of components as in the random sampling study. However, protein

analysis using TCA precipitation indicated that only 30% of the Kjeldahl nitrogen was present as true protein. Thus, recovery of a protein by-product from these waters is not likely to be feasible. Variability in component concentration from sample to sample was notably high, indicating the lack of fine control, and high variability in processing.

The rejection of COD in the ultrafiltration trials ranged from 58.1 to 86.8%. This relatively low rejection of organic solutes indicates that ultrafiltration using these membranes would not be an effective method recovering a food or feed by-product from these waters. Permeate waters were found to be free of turbidity and reduced in color intensity. Concentrate fractions appeared viscous in consistancy and sometimes gelled on cooling.

The pretreatment of desludger feedwaters using filtration and activated carbon was found to have limited success in the prevention of membrane fouling. In all trials a gel like fouling layer was found to line the lumen of the hollow fibers. This carbohydrate based foulant was resistant to chemical cleaning and was physically removed using turbulent flushing.

Reverse osmosis of the protein desludger waters yielded concentrate fractions of approximately 75% carbohydrate, 20% ash and 5% protein on a dry weight basis. This fraction may have potential as an animal feed. The resulting RO permeate

waters contained less than 170 mg/L COD and were relatively free of turbidity, color and pea odor. This processed water could be discharged to municipal sewers with no BOD or suspended solids surcharge. However, reuse of this high quality water in plant processes would be a logical choice providing savings in the purchase, heating and pH adjustment of fresh water and decreased charges for effluent discharge.

Reverse osmosis of the pea hull fiber decanter waters produced concentrates containing approximately 60% carbohydrate, 35% ash and 15% protein. Again, the permeate produced was high quality with less than 180 mg/L total solids. Due to the low concentration of solutes found in this feedwater source, partial or total reuse of these waters in plant operations could be initiated with little or minimal treatment.

Permeate flux values were consistantly higher using the Osmonics 192-SR membrane in comparison to the Filmtec TW30-2026 and Osmonic 192-HR membranes. Althought the latter two membranes offer higher solute rejection properties, this level of purification would likely not be necessary for the discharge or reuse of permeated waters. However, difficulties were encountered in the cleaning of all three membranes making the effective removal of foulants a limiting factor in successful industrial scale-up. Prefiltration of all feedwaters to a one to five micron level is necessary to prevent immediate blinding of the

membranes due to suspended solids.

Laboratory recycle studies indicated that minimal change in protein yields can be expected on the reuse of primary and secondary isolation waters.

Floc formation studies using heat showed that short term storage of protein desludger effluents would induce a fine white floc at various temperatures. However, this floc was found to contain approximately 77% protein in contast to the fouling layer obtained from the UF hollow fibers which contained mainly carbohydrate.

6. FUTURE CONSIDERATIONS

1. Sedimentation and cyclonic pretreatment of process streams, containing high levels of suspended solids, should be studied to reduce the blinding encountered with cartridge prefilters.

2. Chemical analyses of RO foulants should be performed to better define methods of membrane cleaning.

3. Enzyme work should be undertaken to determine the effect of amylases in the cleaning of carbohydrate fouled membranes.

4. Studies with inorganic membranes may provide improved cleanability of membranes using clean-in-place systems.

5. Investigations into the use of organic flocculants may be beneficial in the pretreatment of process waters to prevent membrane fouling.

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APPENDIX 1.

Terms and Definitions

1) Feed - Solution which enters the system and is pressurized

2) Permeate - Solution (purified water) which passes through the membrane

3) Concentration (retentate) - The solution which exits from the system which has not passed through the membrane. It is enriched in rejected materials.

4) The percentage of dissolved material which does not pass through the membrane.

$$\text{Rejection} = 1 - \frac{\text{concentration of permeate}}{\frac{\text{conc. of feed} + \text{conc. of concentrate}}{2}} \times 100$$

5) Recovery - The ration of permated rate to feed rate

$$\text{Recovery} = \frac{\text{permeate rate}}{\text{feed rate}} = \frac{\text{permeate rate}}{\text{permeate rate} + \text{concentrate rate}}$$

6) Effective Pressure - Actual pressure available to force permeate through the membrane.

$$\text{Effective Pressure} = \text{Applied (operating) pressure} - \text{Osmotic pressure} - \text{Back pressure}^{1}$$

[1]permeate back pressure is assumed to be 0