TREATMENT OF RICE COOKER WASTEWATER AND RECOVERY OF BY-PRODUCTS BY MEMBRANE MICROFILTRATION

J. C. Boykin, T. S. Soerens, T. J. Siebenmorgen

ABSTRACT. *A pilot scale system was used to evaluate the performance of a membrane microfiltration system, rated at 0.1 micron, for the recovery of starches and other solids from rice cooker wastewater produced by a rice processing facility. The first series of tests revealed that a crossflow velocity (V) of 5 m/s and a transmembrane pressure (TMP) of 275 kPa were appropriate for filtration. In the next series of tests, a concentration of almost 9% total solids was achieved, and the logarithmic relationship between permeate flux and concentration was described. Permeate flux rates decreased from over 80 L/m^2*hour (LMH) for unconcentrated samples to under 20 LMH for the most concentrated samples. The feed flow contained an average of 0.71% total solids and 4395-mg/L biochemical oxygen demand (BOD). The permeate contained under 0.10% total solids and 330 mg/L BOD. A membrane system was sized for the test site based on data from these experiments. A system capable of concentrating 68 L/min of cook water to 5%, 8%, and 10% total solids required a total membrane surface area of 75, 100, and 115 m^2, respectively. The reduction in sewage expense was predicted to average $10.48 per hour of operation.*

Keywords. *Cooked rice, Wastewater, Membrane, Filtration, Microfiltration.*

In some convenience food plants, rice is cooked before it is added to the final product. After cooking the rice, the very starchy process water is often disposed at a cost as wastewater. One plant reported having a chemical oxygen demand (COD) of up to 20,000 mg/L and a BOD of up to 10,000 mg/L (Stover, 1992). The solids from this type of wastewater can be hard to settle out, making it difficult to treat (Potter and Hensley, 1999).

Biological processes can be used to pre−treat starchy wastewater and minimize sewage output. With waste streams high in suspended solids and organic matter, anaerobic biological treatment systems are preferred to aerobic processes (Flaherty and Smick, 1989). For anaerobic systems, sludge production is less and biogases can be recovered and utilized. The downfalls associated with anaerobic systems are that they produce more nuisance odors, are more sensitive to cleaning agents, and must adhere to stricter environmental guidelines. Different anaerobic biological treatment processes have proven effective in treating wastewaters high in carbohydrates. Studies have been conducted on corn (Kwong and Fand, 1996), wheat (Flaherty and Smick, 1989), potato (Koster and Lettinga, 1985), and rice wastewaters (Stover, 1992). These systems have proven to reduce COD and BOD by nearly 100% when operating under optimal conditions.

There has been a recent shift from processes that dispose of waste to processes that recover part of the waste as a valuable by-product. Processes have been developed to concentrate starch from waste streams by techniques such as evaporation and spray drying (Strolle et al., 1980). The recovered starch can be land applied for disposal or can be used more profitably as animal feed, fermentation medium, or human consumption.

Membrane filtration has become increasingly popular in by-product recovery and can be used to concentrate or purify a solution (Koseoglu et al., 1991). Wastewater treated with membrane filtration does not require much additional treatment (Potter and Hensley, 1999). In high pressure, tubular-type membrane filtration, crossflow filtration occurs (fig. 1) where the flow direction of the solution is parallel to the filter surface (Koseoglu et al., 1991). A pump is used to pass a solution through a membrane system under pressure. Some of the solution passes through the filter as permeate, and the rest of the solution continues along the surface of the filter as concentrate. The solution permeating the membrane contains particles small enough to pass through the membrane. Typically, the solution recycles through the system, with the flow rate within the system much larger than the flow rate into the system.

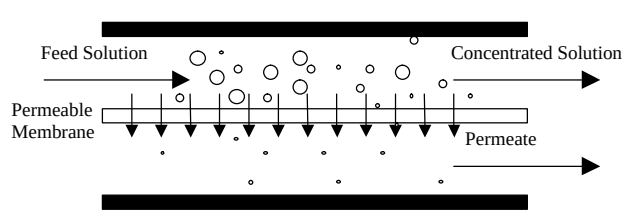

Figure 1. Crossflow membrane filtration diagram.

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The authors are **James Clifton Boykin,** Agricultural Engineer, U.S. Cotton Ginning Laboratory, Stoneville, Mississippi; **Thomas S. Soerens,** Associate Professor, Department of Civil Engineering, University of Arkansas, Fayetteville, Arkansas; and **Terry J. Siebenmorgen, ASAE Member Engineer,** Professor, Department of Food Science, University of Arkansas, Fayetteville, Arkansas. **Corresponding author:** J. Clif Boykin, U.S. Cotton Ginning Laboratory, P.O. Box 256, Stoneville, MS 38776; phone: 662-686-3097; fax: 662-686-5483; e-mail: cboykin@ars.usda.gov.

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Membranes are fouled when particles in the feed solution adsorb to or collect on the membrane surface (Brockman and Seyfried, 1997). This layer of materials thickens as the system is operated and reduces permeate flux. This layer is continuously removed by the tangential flow of turbulent solution across the layer and is deposited as particles are separated from the permeate. The accumulation of particles is at first rapid but quickly stabilizes with further accumulation very slow. Operating at high flow velocities helps remove particles from the surface of the membrane and the TMP is the driving force of permeate flux, but there is an optimum setting for both where further increases do not increase permeate flux. Permeate flux also decreases as the concentration of the feed solution increases.

Rice starch granules ranges in size from 3 to 5 μm, but fragments resulting from cooking could be much smaller (Singh et al., 2003). A 0.1-μm membrane filter was evaluated for concentrating starches in the effluent from a large rice cooker. The concentrated starch solution could be a valuable by-product and the filtered water could be discharged at a reduced cost. The objectives were to determine the optimal V and TMP, the properties of the recovered product, and the properties of the purified process water. The size and cost of the system and the sewage savings were also reported.

Materials and Methods

Filtration Test Unit

A DS Scepter membrane test unit (model number DS2, Graver Technologies Inc., Glasgow, Del.) was used to evaluate the feasibility of separating starches from rice cook water using membrane separation. The membranes were tubular and constructed of stainless steel with a titanium dioxide lining. They were chosen for this experiment because of their durability. They were rated to pressures over 2000 kPa, temperatures over 120°C, and both high and low pH needed for cleaning.

Figure 2 is a schematic of the test unit. The stainless steel tubular membranes (module P/N 2.5-750A-5P) were rated at a pore size of 0.1 micron in diameter. There were three modules connected in series, with each module consisting of three membranes in parallel within an outer shell. The length of each membrane was 1.52 m, and the inside diameter of each membrane was 1.83 cm. Each module had a tube and shell structure; three smaller membrane tubes within a larger shell cylinder. The permeate passed through the walls of the membrane tubes and collected in the shell of the module. The total inside surface area of the membrane tubes was 0.788 m^2.

The modules were connected in series with a heat exchanger, which used steam to maintain the fluid temperature within the system. The heat exchanger consisted of four tubes within an outer shell. As the feed solution flowed through the four tubes, steam entered the top of the shell and passed across the surface of the tubes heating the fluid. A 230-L insulated tank was used to store the fluid pumped through the system. A positive displacement feed pump (FP) coupled with a 2.24-kw (3-hp) motor (model M3611T, Baldor Electric Co., Fort Smith, Ark.) pumped fluid from the tank into the system and pressurize the system, while a Waukesha centrifugal circulation pump (CP) coupled with a 3.73-kw (5-hp) motor (model JMM3613T, Baldor Electric Co.) circulated the fluid within the system. Pressure within the membrane was regulated by a ball valve, which restricted the flow of concentrated fluid leaving the system. Flow velocity through the membrane was regulated by a butterfly valve that restricted the flow of fluid entering the modules. There was a temperature and a pressure gage at the entrance of the heat exchanger, and a pressure gage at the outlet of the modules. There was a flow meter for fluid circulating within the system, and flow rates exiting and entering the system were determined manually.

The system was cleaned after each day of use. Sodium hydroxide solution was heated to near boiling and circulated through the system for about an hour. A cool water phosphoric acid solution with detergent was then passed through the system to remove caustic residue. Clean water flux rates were checked to ensure complete cleaning.

Test Site

The tests were conducted at the Entrée plant of Tyson Foods Inc. in Fayetteville, Arkansas. This plant was equipped with a rice convenience cooker capable of cooking up to 3600 kg (8000 lb) of uncooked rice per hour. The rice was cooked semicontinuously. Rice entered the cooking cycle and was pushed through the cook water with an auger. Next, the rice made a similar pass through cooling water. The rice leaving the cooling cycle was drained and packaged. The cycle of rice flow into the cooker was 20-min continuous flow followed by 5-min no flow.

The cook water was heated to about 82°C by steam injection. The cook water left the cooker at about 68 L/m. Based on preliminary results, the cook water had a much higher BOD and solids content than the cooling water, and the cook water was already at very high temperatures. For these reasons, only the cook water was filtered.

Procedures

In the first series of tests, appropriate settings for V and TMP were established to maximize flux. Cook water was strained with an ordinary kitchen strainer and added to the holding tank directly from the outlet of the cooker. The temperature of the cook water (82°C) was maintained in the filtration system with steam. The concentration of the fluid was maintained by returning the permeate to the holding tank. The filter was operated at several combinations of TMP and V, starting at the lowest pressure. The system was allowed to stabilize for 10 min at each setting before taking measurements or samples. The system was cleaned and the process repeated a total of three times.

In the second series of tests, the effect of solids concentration on filter performance was studied. The previously described procedure was used, but the TMP was maintained at 275 kPa and V decreased from 5.5 to 4.0 m/s through the test due to the system's pumping limitations. The permeate was removed from the system in this second part of the test allowing the concentration of fluid in the system to increase. The solution was concentrated for about 7 hours during each test until the flux rates were greatly reduced. Flow measurements were taken for permeate, concentrate returning to the holding tank, and concentrate returning to the system. Pressures and temperature were recorded and samples of permeate and concentrate were collected. The procedure was repeated twice. Nine samples from both

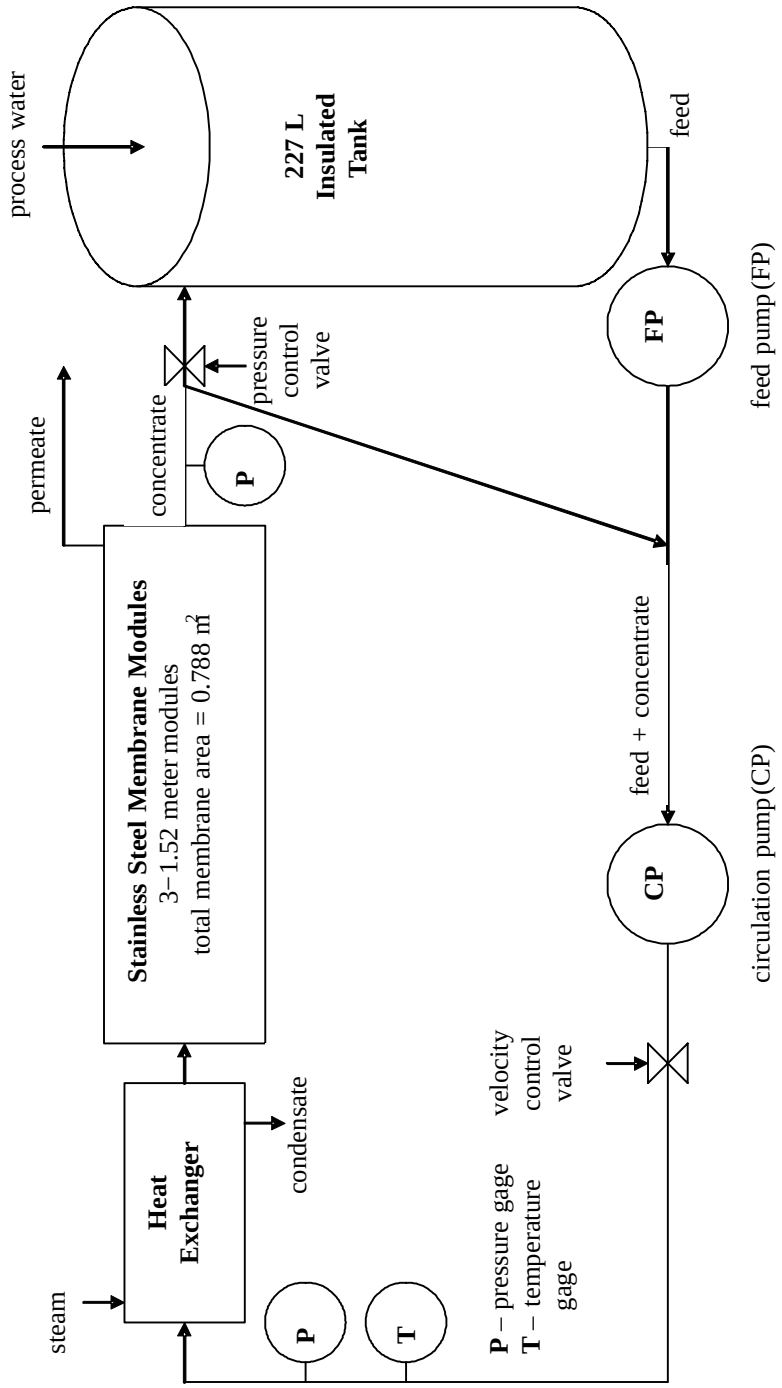

Figure 2. Schematic of the membrane filtration test unit.

replications were analyzed for total solids concentration. Some of the samples from the second replication were also analyzed for BOD, protein content, and carbohydrate content.

Statistical analysis and model development were done using the SAS mixed analysis procedure (SAS, 2001). The levels of significance for model parameters were reported with the results.

Sample Analysis

Total solids concentration of the samples was measured according to method 209 (APHA, 1980). Total suspended solids (TSS) were determined by the same method after filtering the sample through a 0.45-micron glass fiber filter.

The BOD of the samples was measured according to method 507 (APHA, 1980). The samples were diluted with BOD dilution water and seed solution was added. Either 2 or 3 mL of plant influent to the Fayetteville, Arkansas wastewater treatment plant was added as seed material, and BOD results were corrected for the strength of the seed.

Crude protein of the samples was determined by method AOAC 990.03 (AOAC, 2000) utilizing a Fisons NA2000 for combustion after evaporating samples in an oven at 103°C for 48 h. Methods used by Wang and Wang (2000) were followed for the analysis of carbohydrates. All samples were analyzed

with a Waters system high-performance size-exclusion chromatograph (HPSEC) (Waters Corp., Milford, Mass.). Sugars with a degree of polymerization (DP) of 1 to 79 were used along with Dextran standards including amylose and amylpectin ranging in average molecular weight from 5,200 to 872,300 as standards in HPSEC analysis (Sigma, St. Louis, Mo.).

Results and Discussion

System Stability

The system stabilized within 5 min after adjusting V and TMP settings. Measurements for each treatment were collected after allowing 10 min for the system to stabilize. Flow velocity varied by 0.03 m/s and flux rates varied by 1.2 LMH. Temperatures were controlled within 1°C of the set temperature and pressures were controlled within 7 kPa.

Changes in Flux with Flow Velocity and Transmembrane Pressure

Flux appeared to increase with TMP, but increases above 275 kPa were minimal and seen in only one test. Throughout the tests, flux appeared to increase up to the maximum V of 5 m/s. The data from each test were combined to develop a model for permeate flux rate with V and TMP.

$$\text{Flux} = -73.8 + 56.3 \ln(V) + 15.7 \ln(\text{TMP}) \qquad (1)$$

where

flux = permeate flux rate (LMH)
V = cross flow velocity (m/s)
TMP = transmembrane pressure (kPa)

The significance levels (p-values) of the parameters in equation 1 were 0.014 for the intercept and under 0.001 for the two parameters associated with V and TMP. Since energy inputs are proportional to TMP and V^2, flux was plotted with V^2 at TMP = 275 (fig. 3) and with TMP at V = 5m/s (fig. 4) based on the model parameters (eq. 1). At these settings, decreasing returns in flux rates were seen with increases in V^2 (fig. 3) and TMP (fig. 4). Equation 1 was used to predict a flux rate of 105 LMH at 275 kPa and 5 m/s. A 1% increase in TMP caused flux rates to increase 0.15%, and a 1% increase in V^2 caused flux rates to increase by 0.27%, so further increases in TMP or V caused minimal changes in flux rates. A TMP of 275 kPa and V of 5 m/s were found to be appropriate for the tested filter (0.1 μm) in rice cook water filtration.

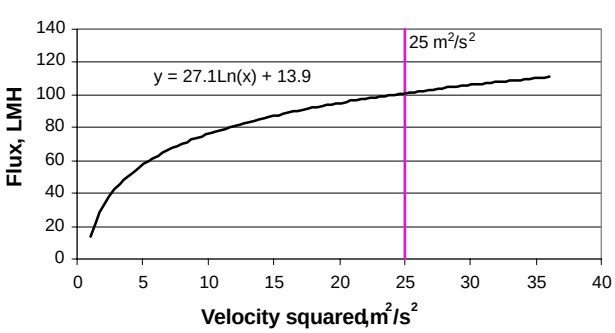

Figure 3. Plot of flux rate with V^2 at TMP = 275 kPa based on regression model parameters.

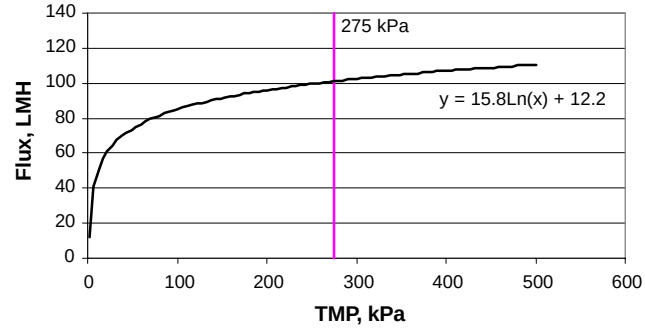

Figure 4. Plot of flux with TMP at V = 5 m/s based on regression model parameters.

Changes in Flux with Concentration

The decline in permeate flux was primarily due to increased solids concentration, though the decrease in V had some effect. Permeate flux decreased logarithmically with total solids concentration (C_F) from nearly 125 to less than 20 LMH as C_F increased from under 1% to 8%. The data from the two tests were combined to model the influence of C_F on flux rates.

$$\text{Flux} = 77.9 - 28.0 \ln C_F \qquad (2)$$

where C_F = total solids concentration of the feed (%)

The significance levels of the parameters in equation 2 were less than 0.0001. These parameters are only valid for the conditions described for this test system.

Analysis of Solids Content

The concentration of solids in the unfiltered cook water was measured for 10 samples that covered all stages of cooking, from rice entering the cook cycle to filling the cook cycle to emptying the cook cycle. Total solids averaged 0.71% and ranged from 0.32% to 1.05% with a standard error of 0.061. Total suspended solids (TSS) was measured for three samples and ranged from 43% to 49% of total solids. Seven of these samples were also analyzed for BOD. There was a strong positive correlation ($R^2 = 0.98$) between BOD and total solids, and each percent total solids equaled 6200 mg/L BOD. For two samples with high solids content, BOD was not measured. Based on the range of solids content, the BOD of unconcentrated cook water ranged from 1981 to 6499 mg/L and averaged 4395 mg/L.

The concentration of total solids in the permeate was measured as the feed was concentrated in two tests. There was a slight increase in the permeate solids content with feed concentration in one test, but no trend was seen in the other test. Overall, 17 permeate samples averaged 0.0734% total solids and ranged from 0.036% to 0.118% with a standard error of 0.0073. Three permeate samples analyzed for BOD. These values were 153, 377, and 300 mg/L BOD.

An unconcentrated (UC) sample was analyzed for its carbohydrate profile (table 1). Also analyzed were corresponding permeate and concentrate samples, identified by the solids concentration of the concentrate, for example, the labels “P, 1.90%” and “C, 1.90%” refer to the permeate and concentrate samples, respectively, taken when the total solids concentration of the concentrate was 1.90%. Carbohydrates in the samples analyzed were categorized and quantified according to their degree of polymerization (DP) of glucose.

Table 1. Percent distribution of carbohydrates for the feed sample and for paired samples of permeate and concentrate.

Sample	DP=2	DP=3	DP=4	DP=16	DP=79	Amylose	Amylopectin
UC[a]	27	0	0	53	0	7	12
P[b], 1.90[c]	28	0	38	35	0	0	0
P, 3.82	35	0	27	38	0	0	0
P, 8.54	31	0	22	35	11	0	0
C[b], 1.90[c]	6	11	0	0	0	18	66
C, 3.82	1	3	0	0	0	18	78
C, 8.54	0	0	0	0	0	18	82

[a] UC was an unconcentrated feed sample.
[b] P and C were permeate and concentrate samples.
[c] P and C were taken in pairs as C was concentrated. This number is the solids concentration of the concentrate.

Amylose (DP = 1500 to 6000) and amylopectin (DP = 300,000 to 3,000,000) were the two large carbohydrate components of rice starch.

It can be seen in the carbohydrate profile that none of the larger molecules, amylose and amylopectin, were present in the permeate. These molecules were too large to pass through the membrane's pores. As the solution became more concentrated, there was more amylose and amylopectin present in the concentrate. Many of the smaller carbohydrates, with a low DP, escaped from the concentrate into the permeate stream.

Crude protein was analyzed for one feed sample (unconcentrated screened cook water) and one sample of final concentrate. Protein contents were 0.07% and 0.45% for feed and concentrate, respectively, when solids concentrations were 0.92% and 7.30%. The ratio of concentrate to feed was 6.43 for protein and 7.93 for solids. These ratios were similar, indicating that the filter retained most of the protein.

Savings in Sewage Expense

Solids and BOD properties of the feed and permeate were used to estimate the sewage cost savings for pre-treating cook water by membrane filtration. The flow rate leaving the rice cooker averaged about 68 L/min (18 gpm). The weight of BOD and TSS removed can be determined by multiplying the reduction in their concentrations by the flow rate of the process water. Removal of BOD ranged from 7 to 25 kg/h. No suspended solids could pass the filter, so 6.5 to 21 kg/h TSS were removed. The surcharges from the waste treatment plant were \$0.4544/kg BOD and \$0.2271/kg TSS (Fayetteville, 2001). Reductions in sewage cost by filtration ranged from \$3.12 to \$11.49/h for BOD and from \$1.48 to \$4.86/h for TSS with an overall average of \$10.48/h.

System Scale-Up

Sizing the full scale system was done by determining how much membrane area was required to filter the cook water to a particular concentration.. This system was to include three components for three stages of filtration. Components were to be the same size, so they were not stage specific. One equation needed to size the system was the solids balance for each step:

$$Q_C \times C_C = Q_F \times C_F - Q_P \times C_P \quad (3)$$

where

Q_C = the flow rate of concentrate (L/h)
C_C = total solids concentration of the concentrate (%)
Q_F = the flow rate the feed (L/h)
C_F = total solids concentration of the feed (%)
Q_P = the flow rate the permeate (L/h)
C_P = total solids concentration of the permeate (%)

The concentrate flow rate (Q_C) was the difference between the feed (Q_F) and permeate (Q_P) flowrates:

$$Q_C = Q_F - Q_P \quad (4)$$

Equation 4 was used to eliminate Q_C from equation 3. Parameters used in the design were a maximum Q_F of 76 L/min, maximum C_F of 1%, and minimum C_P of 0.07%. So, Q_P can be solved from C_C. Equation 5 related the required membrane surface area (A) to flux rate (F) and permeate flow rate (Q_P):

$$A = Q_P / F \quad (5)$$

where A = membrane surface area (m^2)

For a one stage system, Q_P can be solved for the desired C_C, (eq. 3 and 4.), and F can be solved for the desired C_C (eq. 2). Then, equation 5 can be used to solve for A. For a three stage system, only the final desired C_C is known. This value was approximated for the first and second stage to solve for Q_P and Q_C. The Q_C and C_C for one stage were the Q_F and C_F for the next stage. An iterative process was used to equate A in each stage by changing C_C for the first two stages. The system designed to achieve a final total solids concentration of 5%, 8%, and 10% required 75, 100, and 115 m^2 of total membrane surface area, respectively. Representatives of Graver Technologies Inc. (Glasgow, Del.) estimated the budget cost for the commercial scale membrane system to range from \$550,000 to \$990,000.

Conclusions

Rice cook water, high in carbohydrates of starch and degraded starch, was concentrated using a 0.1-micron tubular microfiltration test system. The logarithmic increase in flux with both TMP and V was described in one series of tests, and an appropriate TMP (275 kPa) and V (5 m/s) were determined for filter operation. The logarithmic decrease in flux with solids concentration was described in another series of tests at 275 kPa and 5 m/s as solids concentration increased from 0.7% to over 8%.

The purified water (permeate) removed from the system contained no suspended solids and was 90% to 95% lower in BOD than the feed. The solids content of the permeate was under 0.1%, including some small degraded starch fragments but no starch or protein. Comparing the disposal of permeate to the original feed stream, the predicted savings in sewage expense by removing solids and BOD ranged from \$4.60 to \$16.35/h of rice cooker operation, depending on the strength variations of the cook water.

For the rice cooker wastewater in this experiment, a three-stage system capable of treating 68 L/min would require 75, 100, and 115 m^3 of membrane surface area to achieve a product concentration of 5%, 8%, and 10%, respectively. Representatives of Graver Technologies Inc. (Glasgow, Del.) estimated the budget cost of this system to range from \$550,000 to \$990,000.

For an average savings in sewage expense of \$10.48/h, the yearly savings was calculated for 72 h of weekly operation to be \$39,000. The present value of annual payments of \$39,000 at a yearly interest rate of 8% was calculated to be \$270,000 after 10 years and \$400,000 after 20 years, so savings in sewage expense alone did not justify the cost of the system.

In order to justify the installation of this system at the facility, either a valuable use of the recovered starch would have to be available or increases in sewage fees would have to be seen. Less costly systems may also be evaluated for the same purpose.

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