9-1-2000

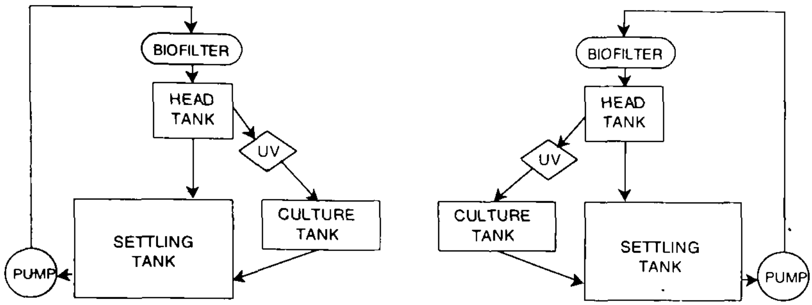
TB177: Evaluation of an Experimental Filter Medium for Water Re-use Systems

John Riley

David Cole

Robert Bayer

ISSN 1070-1524



Evaluation of an Experimental Filter Medium for Water Re-use Systems

John Riley

David Cole

Robert Bayer

Technical Bulletin 177 **September 2000**

MAINE AGRICULTURAL AND FOREST EXPERIMENT STATION

ne

Evaluation of an Experimental Filter Medium for Water Re-use Systems

John Riley
Professor

David Cole
Graduate Student

Robert Bayer
Professor

Department of Biosystems Science and Engineering
University of Maine
Orono, Maine 04469

INTRODUCTION

This report describes the development and testing of a new material for use as a filter medium in recirculating aquaculture systems and other water quality control situations. The material consists of a combination of activated carbon and a hydrophilic urethane foam in a homogeneous matrix. Its physical properties relevant to filtration were determined. It was then tested for its ability to remove various dissolved organic compounds and inorganic nitrogenous compounds in the form of a synthetic waste-water containing ammonia and nitrite. Finally it was subjected to long-term use in the biofilter of a fish culture system. It proved to be a successful filter medium for several applications and showed great potential for use in biofilters installed in recirculating aquaculture systems and other fish and shellfish culture facilities.

BACKGROUND

In 1991 research was being conducted at the University of Maine into the development of an activated carbon filter medium for removing traces of gasoline in well water. Activated carbon powder and granules are effective in removing organic compounds, but can be dusty, messy, and generally inconvenient. A local manufacturer had recently developed a patented hydrophilic polymer foam, which was being sold as pre-soaped sponges, foot odor absorbers, and other products. In a collaborative research project with the company, it was found that the polymer could be combined with the finest activated charcoal powder such that the powder was completely and permanently distributed and bonded within the foam. It became apparent that the material had potential as a filter medium not just for chemical treatment but as a physical particulate filter with applications in fish and shellfish culture systems.

Over the next few years the filter foam was manufactured and sold for use in large commercial lobster holding systems throughout New England. It has proven to be very successful and in some installations was used continuously for three years in systems holding more than 20,000 lbs of lobster. The water quality goals in these short-term holding systems were clarity through removal of silt and organics, and reduction of odor and off-taste in the product. Little or no thought was given to biofiltration and build up of nitrogenous compounds as the lobsters were not being fed and the water was kept at a very low temperature to reduce the metabolism of the animals. Eventually it became obvious that the material offered several apparent advantages as a bio-filter medium including low density, very large surface area, and convenience in handling, as well as making a separate carbon filter unnecessary. With the growing interest in a more rigorous, engineering approach to recirculation system design, a decision was made to initiate research aimed at collecting and publishing data on the physical characteristics of the material and its performance as a chemical and biological filtration medium.

EXPERIMENTAL DESIGN

The research was divided into three separate and distinct parts: (1) physical, (2) chemical, and (3) biological. The physical properties of interest for a filter material are bulk density and flow rate/pressure drop characteristics as well as the more general properties of ease of handling and cleaning, and useful life. The chemical properties are related to the performance of the activated carbon within the matrix in removing various organic compounds from the waste stream, which in a fish culture system result in off flavors in the product. Biological filtration properties refer to the performance of the material as a biofilter medium in providing a surface for the attachment, growth, and multiplication of nitrifying bacteria necessary to remove inorganic nitrogenous compounds from the water, particularly ammonia and nitrite both of which can be toxic to the cultured species

Physical Properties

Determination of the physical properties was relatively straightforward. Samples of the material were made using different proportions of polymer, carbon, water and surfactant. Although there is the possibility of almost unlimited combinations of ingredients and more than 30 different carbon grades available from a single manufacturer (Nebey-Cheney, Columbus, OH), for practical reasons two grades of carbon and two ingredient combinations were used. These were

Carbon Grades

Type UU	50 X 200 mesh
Type YF	Powder (through 325 mesh)

Ingredient Combinations (by weight)

Polymer:Carbon:Water	3 : 1 : 2
	4 : 1 : 2

When the liquid ingredients are combined there is a rapid, exothermic reaction and expansion into a foam, which becomes solid within seconds. Changing the ratio of the ingredients affects the expansion rate, which in turn affects the density and porosity of the foam. The samples were poured in cylindrical containers so calculation of bulk densities consisted simply of weighing and measuring the solidified foam. The temperature of the ingredients was kept constant.

Flow rate/head loss characteristics were determined by placing the samples in a 150-mm-diameter cylinder with a perforated bottom. Water was pumped in from the top and allowed to flow through the material. Varying flow rates were applied and inlet pressures measured with a dial pressure gauge establishing a flow rate/head loss curve for the sample.

Chemical Filtration

Filtration with activated carbon works through adsorption, a physical action based on molecular forces. When carbon is activated, millions of holes and interconnected passages are created, resulting in a tremendous surface area. When a liquid containing impurities comes in contact with the carbon, the impurities are attracted and held on these internal surfaces to a degree dependent on the relationship between the size and the shape of the contaminant molecules and the size and extent of the carbon pore structure. Many substances can be and are removed from water using activated carbon, and it has been used in water purification for many years. In relation to fish and shellfish recirculation systems, it has a high capacity for removal of organic compounds causing discoloration, off-odors, and off-flavors, but has a variable capacity for removing different nitrogenous compounds.

The first experiment was conducted to determine the relative filtering capacity of the carbon foam mix and the carbon alone. Batches of both materials were placed in equilibrating bottles and challenged with two different doses of chlorobenzene (an organic compound used as a standard for evaluating carbon filtering capacity), kept at constant temperature for six hours after which the final concentrations of the organic were measured. Additional tests were done on the ability of the carbon foam mix to remove chlorine, gasoline, riboflavin, and ammonia by making up mixtures and solutions at a known concentration, passing it through the filter, and measuring the residual concentrations.

Biological Filtration

While the above experiments were relatively simple and could be completed in a few days, measurement of biofiltration performance is difficult and requires long-term experiments, as colonization of a medium with nitrifying bacteria can easily take several weeks. Although some idea of biofilter performance can be obtained using an artificial wastewater, for a more realistic picture it is necessary to conduct at least some of the work using a live fish culture system. The type and level of nitrogenous wastes generated in a fish tank can be quite variable, depending on feed rate and content; feed consumption rates are to some extent dependent on the stress levels of the fish, and these stress levels in turn can be affected by water quality. Thus there is potential for an extremely dynamic situation.

Atlantic Salmon (Salmo salar) was the species chosen for these experiments based on previous experience with this species and its commercial importance in Maine. Two parallel, identical recirculating systems were designed and constructed; this allowed for experimental repetition and measurement of the effects of varying one component or condition in one system while maintaining the other as a control. Each system consisted of three cylindrical fiberglass tanks arranged as shown in Figure 1. The culture tank was of 350 L capacity, with a standpipe outlet draining into a 1000-L settling tank. From the settling tank, the water was pumped by a 1-kw centrifugal pump through the biofilter from where it trickled into the 350-L head tank. A standpipe in the head tank maintained a constant flow to the culture tank with the overflow spilling into the settling tank below. An adjustable bypass valve allowed for a proportion of the water to be directed around the biofilter directly into the head tank. A separate cooling loop was provided from the head tank, through a 200-watt chilling unit, and back to the head tank. A UV sterilizer was placed in the line between the head tank and the culture tank. All piping was PVC, with flow rates controlled by ball valves. High/low float switches in the culture tank shut down the circulation pump to prevent draining or overflow of the tank.

The biofilter was a simple trickling filter and consisted of a PVC box containing the medium to be tested. Each filter box had a perforated bottom on which was placed 40 mm of crushed limestone covered with fiberglass window screening, then the biofilter material, and finally a layer of Dacron fiber to screen out any large particles. The material for all of these tests was poured in blocks in a mold and then reduced in size to cubes approximately 50 mm on the side. The inflow to the filter was dispersed evenly over the medium with the treated water draining through perforations in the floor of the filter box and into the head tank.

Artificial seawater was used for all the experiments and was maintained within a salinity range of 28–33 ppt and a temperature range of 8–9°C. Daily water quality measurements were taken for total ammonia-nitrogen, nitrite-nitrogen, salinity, temperature, pH, and dissolved oxygen. Nitrate-nitrogen levels were measured once per week. Ammonia, nitrite, and nitrate concentrations were measured colorimetrically with a LaMotte Smart Colorimeter (Chesterville, MD). Salinity was monitored with a handheld Pinpoint meter (American Marine, Inc.) supplied by Aquatic Eco-systems, Inc. (Apopka, FL), pH with a Cole-Parmer pHtestr2 (Vernon Hills, IL), and dissolved oxygen with an Otterbine Sentry 3 meter (Emmaus, PA). A water change of approximately 5% system volume was carried out weekly, with the water being drained from the bottom of the settling tank so as to remove any sediment therein.

Three separate experiments were conducted on the biofilters. The first experiment was done to show the response of the filter foam to a challenge from chemically synthesized wastewater, in

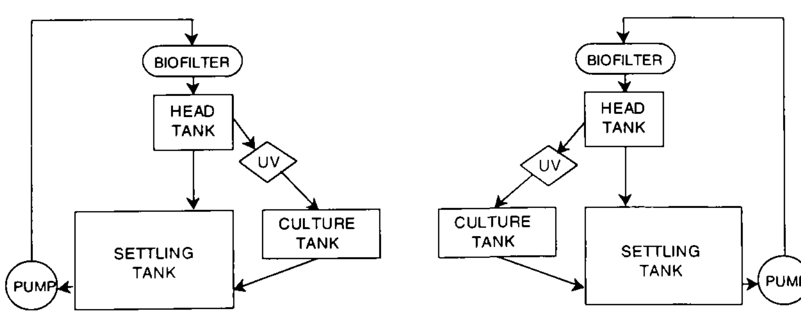


Figure 1. Schematic diagram of parallel recirculation systems

order to determine if in fact the material would support a population of nitrifying bacteria. Fifty grams of ammonium chloride and 23 g sodium nitrite were added to each system. The biofilter medium was inoculated with a small amount of material from a different filter in another active fish culture system. This experiment was run for nine days and the water quality parameters as listed in the previous paragraph were measured.

The second experiment was conducted with live fish. The first objective was to test the material under realistic conditions and to determine the maximum capacity of a specific amount of material, i.e., the greatest amount of biomass the system could sustain while still maintaining acceptable water quality. A second objective was to see whether conventional design procedures can be used to predict the amount of biofilter material needed for a particular situation. These procedures begin with the calculation of the amount of ammonia that will be produced each day. This figure is a function of the protein content of the feed, the nitrogen level in the protein, the protein use factor, and the percentage of total nitrogen excreted as ammonia. There are several different versions of this function (Speece 1973; Lovell 1989; Lawson 1995). For this particular experiment, with the known maximum biomass, and the feed content and rate, the production rates varied from 2.25 to 3.3 g NH^3-N/day; the higher figure was used. Nijhof and Bovendeur (1990) reported that mature fixed film bioreactors in seawater can achieve a maximum ammonia removal rate of 0.28 g/m^2/day. This results in a figure of 11.8 m^2 of surface area needed. Estimating the surface area of the foam pieces, the appropriate volume of foam was put into the filter box. The flow rate per unit of volume through the filter was 660 m^3 m^{-3} day^{-1}. At week 1, each system was stocked with five fish averaging 530 g each. At week 3, five more fish were added to each system, and this procedure was repeated every week up to the seventh week to give a total of 30 fish per system. Feed level during this time was kept constant at 0.5% bodyweight. The feed was metered out by hand so that feeding behavior could be observed and to make sure that all the feed was consumed. At week 8 the feed rates were increased until ammonia levels reached 1.0 mg/L. At this point feed level was kept constant for two days and then reduced by half for the remaining weeks of the trial.

The final experiment involved comparing the performance of the experimental material with a commercially available medium that had a known, or at least advertised, potential. The two systems were equipped and operated identically with one biofilter containing filter foam and the other the commercial material as a control. For a more realistic comparison, the amounts used were again based on surface area available for attachment of nitrifying bacteria. For the commercial product, this indicated a volume of 46 L. The filter foam pieces had a surface area approximately three times that of this product, so for this experiment a volumetric ratio of 3:1 was used. At this point it was still not known whether the bacteria could populate the entire surface area of the activated carbon in the foam or just the macro surfaces of the pieces. The commercial material has non-porous spherical surfaces making it easy to calculate surface area. One system had a 4-inch layer of filter foam, the other system had a 12-inch layer of the commercial product. Both biofilters were then inoculated with approximately 1.5 L of filter medium from an existing, fully functional fish culture system.

This experiment lasted 8½ weeks. At the beginning of week 1, each system was stocked with 10 fish averaging 215 g each. In week 2, 20 more fish were added to each system to give a total biomass of approximately 6450 g per system. The flow rate per unit volume of filter material was 670 m^3 m^{-3} day^{-1} for the commercial product and 2234 m^3 m^{-3} day^{-1} for the filter foam. The fish were fed identical diets at a rate of 2% bodyweight per day. In week 3 feeding was suspended to allow the biofilters to acclimate and then the systems were operated for another 5½ weeks.

Scanning Electron Microscopy

To determine whether the nitrifying bacteria in the biofilters using foam were populating just the surfaces of the individual foam pieces or were in fact present throughout the entire foam matrix, samples of foam from an active filter were sectioned and prepared for examination under the scanning electron microscope.

RESULTS

Physical properties

Ingredient Ratio	Bulk density (kg/m^3)
3 : 1 : 2	96
4 : 1 : 2	104

Flow rate/Head loss

Flow Rate (lpm)	Head Loss (m/cm depth)
0	0
0.25	0.04
0.50	0.07
0.75	0.18
1.0	0.24
1.25	0.28
1.5	0.44
1.75	0.60
2.0	0.77

Chemical Filtration

The table below shows the relative filtering ability of the carbon foam mix compared to the carbon alone. The 4:1 ratio of the doses reflects the 25% carbon content of the mix (on a weight basis).

Material	Dose mg/L	Final Conc. mg/L	Removal Rate mg/gram
Foam/carbon	20	25.3	879
Carbon	5	32.6	442
Foam/carbon	60	4.75	705
Carbon	15	16.2	476

The following table shows the filtering capability of the carbon/ foam mix when challenged with various substances. The figures listed are the maximum initial concentrations that could be removed to a level beyond the detection capability of the test instrument.

	Init. Conc. mg/L	Final Conc. mg/L
Chlorine (free)	14	0
Riboflavin	1000	0
Gasoline	5	0
Ammonia	10	10

Biological Filtration

In Experiment #1, addition of chemical ammonia and nitrite to the un-stocked systems produced typical nitrifying acclimation responses as shown in Figures 2a and 2b. There is an immediate rise in ammonia level on addition of the chemicals followed by an equally immediate reduction at Day 2 reaching a relatively constant level of 0.445 mg/L by Day 5. Nitrite levels showed a similar trend although the rise and fall were somewhat attenuated compared to the ammonia levels.

In Experiment #2 the systems were gradually stocked to the design maximum, 30 fish in each, with increasing feed rates. Figures 3a and 3b show the gradually increasing concentrations of ammonia and nitrite, up to the maximum ammonia level specified, 1.0 mg/L. At this point the biomass density was 73 kg fish/m^3 of culture tank volume with a feed rate of approximately 0.5% bodyweight. The mean maximum weekly ammonia level was 0.782 mg/L although some individual readings reached 1.2 mg/L. Mean maximum nitrite concentration was 1.27 mg/L with some readings over 1.5 mg/L.

The ammonia and nitrite levels during Experiment #3 are shown in Figures 4a and 4b. Even with three times more volume of the commercial medium than experimental material, the trends were similar, although the foam showed a slower activation time for ammonia nitrification. Both systems showed a reduction to tolerable levels of ammonia (0.5 mg/L) by the end of the sixth week. The nitrite concentrations showed no statistically significant differences between the two media.

The scanning electron microscopy performed on the sectioned samples of active filter foam indicated that almost all of the nitrifying bacteria were on the surfaces of the foam pieces with only a few seen within the matrix of the foam.

DISCUSSION AND CONCLUSIONS

The experimental material performed well as a biofilter medium with the additional benefits of providing suspended solids reduction and removal of dissolved organic compounds. It also proved extremely easy to handle, light in weight, and as an activated carbon filter it greatly reduces the mess associated with fine carbon

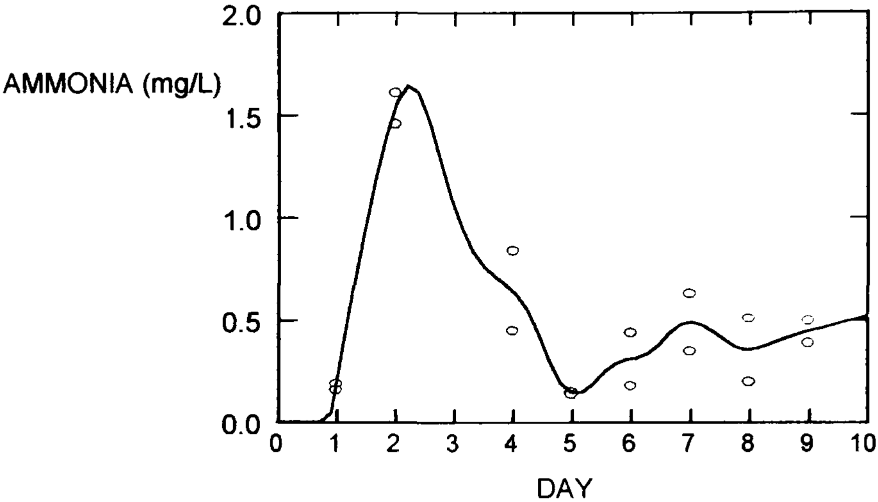


Figure 2a. Ammonia levels resulting from chemical wastewater challenge.

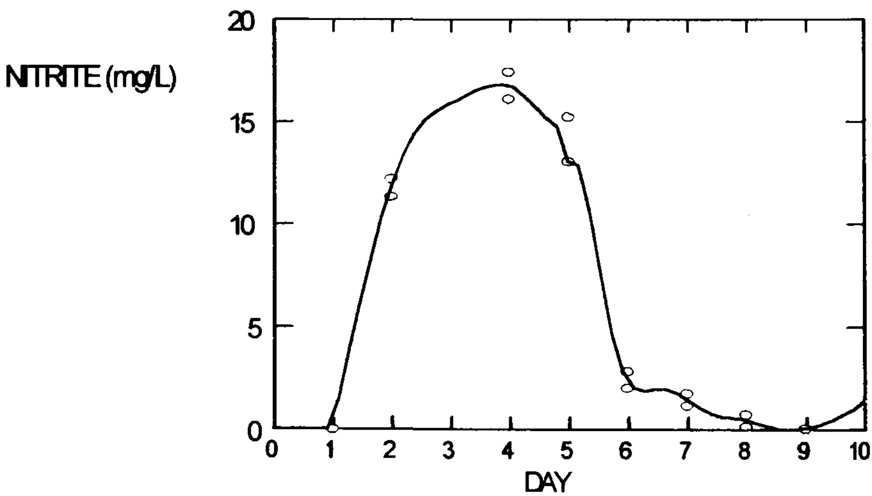


Figure 2b. Nitrite levels resulting from chemical wastewater challenge.

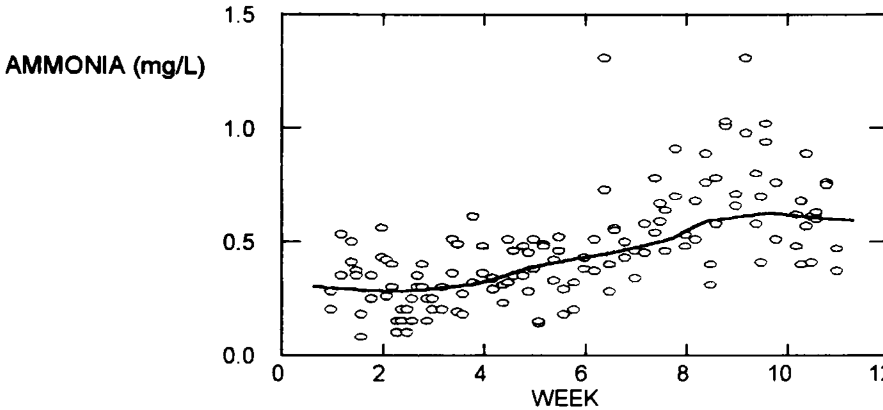


Figure 3a. Ammonia levels resulting from increasing biomass.

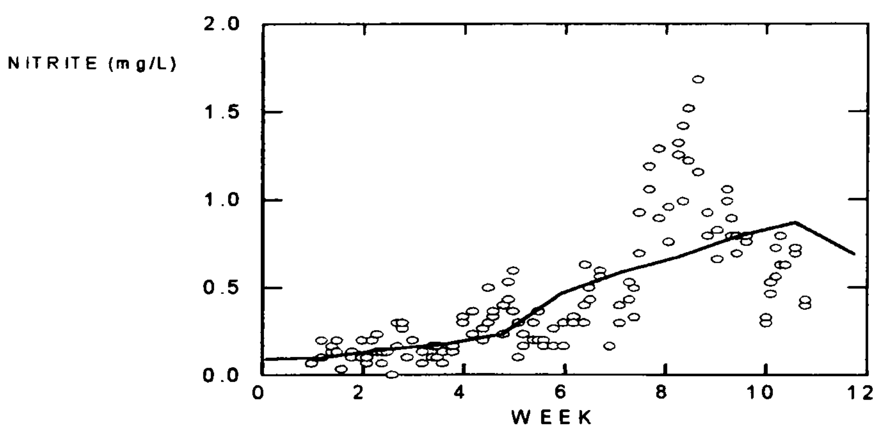


Figure 3b. Nitrite levels resulting from increasing biomass.

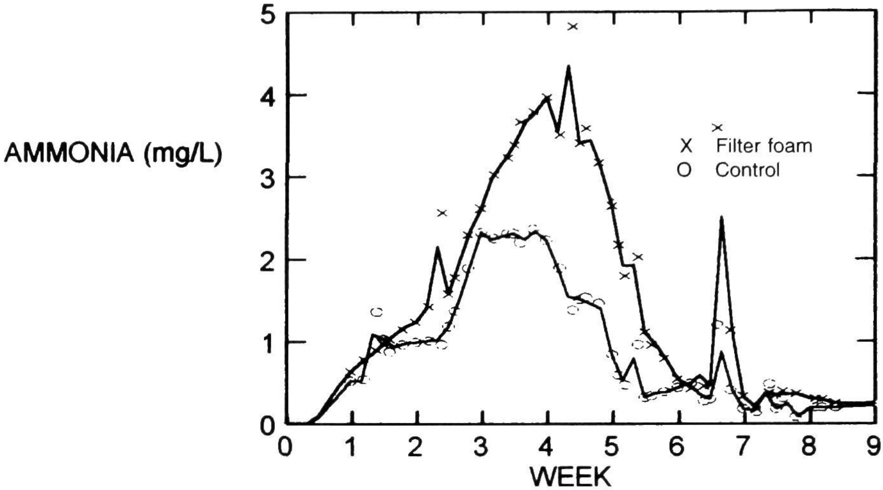


Figure 4a. Comparison of ammonia levels with filter foam and the commercial product

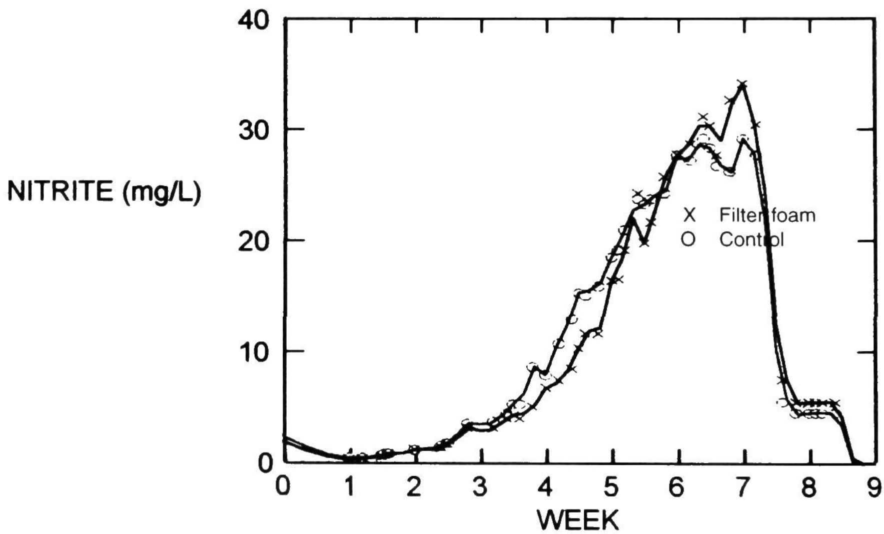


Figure 4b. Comparison of nitrite levels with filter foam and the commercial product

powders. It can be molded or cut to any shape and appears to have an extremely long useful life. Due to its cellular nature it is highly absorbent and remains moist after many days out of water, a factor which would extend the life of the attached nitrifying bacteria in the event of a cessation of water flow through the filter. The ratio of ingredients in the foam does have an effect on the density. Since the polymer is by far the most expensive component, it would seem reasonable to maintain the 3 : 1 : 2 ratio that produced the greatest volume of material for the lesser amount of polymer. As to the carbon type, both of those used produced an acceptable material, but as the finer powder had the greatest surface area it was decided that this become the standard.

Figures 2a and 2b showed the classic response to an artificial, chemical ammonia and nitrite shock loading. The systems quickly recovered by Day 5, indicating the presence of a healthy population of nitrifiers, but it is noted that the ammonia levels rose again after the initial drop, and were still in the 0.4 mg/L range with no biomass present and no further addition of chemical wastewater. One possible explanation for this is that after the initial drop to approximately 0.2 mg/L there was some lysing of the bacteria and the subsequent secondary increase in levels might be attributed to the decaying cellular material from the biofilters. The nitrite concentrations on the other hand steadily decreased to near 0 mg/L by Day 9. This may have been due to the nitrite oxidizing bacteria initially utilizing the nitrite from the ammonia oxidation and then benefiting from the lysed ammonia oxidizing bacteria.

In Experiment #2 the biofilters were gradually challenged in order to determine the maximum ammonia removal rate possible. With all the fish stocked, the system reached equilibrium at a daily feeding rate of 80 g/system. These 80 g of feed are equivalent to 2.76 g of total ammonium nitrogen; with a system volume of 1700 L this represents a daily load of 1.626 mg/L. Figure 3a showed a steady state ammonia level at the end of the experiment of approximately 0.65 mg/L, so the filter was removing 0.976 mg/l/day. This would be an efficiency of approximately 60%, based on the definition that 100% efficiency would reduce the ammonia level to zero. This figure may seem low/ but research by Nijhof and Bovendeur (1990) showed that in a system such as this it is not possible to get the level below 0.25 mg/L.

The final experiment indicated that although the initial ammonia peak was higher for the foam than for the commercial medium, the steady state ammonia and nitrite concentrations were similar and that it was reasonable to equate the nitrifying capacity of the foam as three times that of the other on a volumetric basis. Since the latter have a surface area of 525 m^2 m^{-3}, it would be reasonable to state that the foam has an effective surface area of 1575 m^2 m^{-3}. Although the activated carbon powder within the matrix of the foam has an extremely high surface area, it would seem that this should not be taken into account when designing the biofilter based on surface area, only the surface area of the individual pieces of foam. This seemed to be confirmed by the electron microscopy, which showed a virtual absence of nitrifying bacteria within the matrix. There are at least two possible explanations for this: either there is insufficient flow through the cells of the matrix or there is inadequate dissolved oxygen within the cells, with most of the flow passing over the surfaces of the foam pieces. This would be an argument for making the pieces smaller than the present one-inch size. The value of the carbon is not diminished, however, as it plays a major role in removing organic compounds and associated discoloration of the culture water and off flavors in the fish. Although the filter foam does trap a significant amount of silt and other suspended solids, it is not recommended that it be relied upon as the only mechanical filter if suspended solids levels are high.

REFERENCES

Lawson, T.B. 1995. Fundamentals of Aquaculture. Chapman and Hall, New York.

Lovell, T. 1989. Nutrition and Feeding Fish. Van Nostrand Reinhold, New York.

Nijhof, M., and J.Bovendeur. 1990. Fixed film nitrification characteristics in seawater recirculation fish culture systems. Aquaculture 87:133-143.

Speece, R.E. 1973. Trout metabolism characteristics and the rational design of nitrification filters for water re-use in hatcheries. Trans. Am. Fish. Soc. 2:323-334.