

Article

Treatment and Recycle of Greenhouse Nutrient Feed Water Applying Hydrochar and Activated Carbon Followed by Reverse Osmosis

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Highlights:**What are the main findings?**

- Treatment by self-produced hydrochar (HC) and activated carbon (AC) from tomato plant biomass resolved the issue of toxicity by GNF.
- Other than phytotoxicity from excess ions, no impacts of the pathogens were observed from GNF.

What is the implication of the main finding?

- Higher than the limit concentrations of phytotoxic metal ions in GNF cause impaired plant growth.
- Imbalances of micronutrients in GNFs requires continuous analyses and adjustment.



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Abstract: Leached greenhouse nutrient feed (GNF) water is a great challenge for greenhouse (GH) producers. Unbalanced higher micronutrient metal's phytotoxicity impact GH plant growth, and the high phosphorous levels can cause lake eutrophication if not treated. The analytical results of three GNFs revealed no microbial contamination in any of the GNFs, but the potassium, calcium, magnesium levels, and pH range were above the target level for root zone conditions. Both higher and lower limit concentrations are phytotoxic, causing poor or non-developed roots, leaves, and stems. Sodium was also not in the balanced range. Phosphate and nitrate nutrients were above the measurable range, showing that it would be a threat to lake eutrophication if disposed of. Due to uptake by plants at varied rates, nutrient ion imbalance in GNF is usual, but proper control or treatment is essential as GNF is not a waste but a resource providing fertilization to plants. Potential treatment options investigated include coagulation filtration, sorption with hydrochar (HC), and activated carbon (AC), followed by reverse osmosis (RO) membrane filtration. The HC and AC were produced from waste tomato plants biomass (TPB) of the same GHs to enhance the recycle-reuse of wastes. Neither metals nor nutrient concentrations were reduced to the desired levels by coagulation treatments. The HC and AC treatment provided the recycle-reuse possibility of GNF. RO membrane filtration provided about 97–99% reduction of metals and 99% reduction of nutrients, allowing GNF preparation by adding new fertilizer to the RO permeate. In such a case, the RO reject needs to be reused as feed for TPB carbonization. Different options for GHs to manage TPB and GNF are provided. As RO is an energy-expensive process, an assessment of technical know-how to provide an energy economic process is demonstrated.

Keywords: leached GNF; nutrients; metals; phytotoxicity; eutrophication; reverse osmosis

1. Introduction

Plants grown in greenhouses (GH) gain food nutrients either through moist soil or the circulation of a nutrient-balanced solution (i.e., fertilizer solution), the mostly used method in a hydroponic system. After repeated circulation, part of this GH nutrient feed

(GNF) water is leached for treatment due to imbalances of the micronutrient concentrations from the uptake of required nutrients by plants. Some of the unbalanced ions are toxic to plants, calling for treatment to ensure recycle–reuse. To avoid confusion, it should be mentioned here that the leach GNF is not a wastewater, it is a nutrient resource that needs to be recycled by adjusting or rectifying unbalanced nutrients to enhance the GH economy. Should the GNF quality sufficiently degrade to prevent recycling, the GH can dispose of it by selling to an agricultural agent for application in land crops.

The challenge for the GH owner is confirming to the regulating authority that nutrients are not carried out to the receiving water (Lake Erie) by runoff, as land disposal is regulated to protect Lake Erie from eutrophication. Almost 95% of the GHs in Ontario are located at the southern part of the province, and watershed runoff is discharged into Lake Erie. In 2015, the highest algae bloom in 100 years was observed due to nutrient runoff from both the USA and Canada. This bloom seriously impacted aquatic health, becoming a vital binational environmental concern [1].

Alternative ways of GNF disposal are municipal sewer or handover to a hauler, where, in both cases, GH have to pay huge surcharges on a $\$/\text{m}^3$ discharge basis, including transportation costs. Otherwise, leached GNF is to be stored in a prescribed storage tank, which also incurs huge cost and space for tank building. As a solution to these issues and to provide maximum benefits to this GH economic sector, along with an option for the protection of Great Lake water, the present research was formulated. Overall, the work supports provincial and federal requirements to protect the GH sector, as well as to address the eutrophication issue.

Statistics in 2020 on GH vegetable industries revealed Canada's GHs export of about CAD 1.2 billion, where Ontario's share was about 200 million, and tomato covers the highest part at 20%. Achieving these levels requires efficient water use, which is critical in GH production, especially in some arid climates where the cost of irrigation water exceeds the cost of fuel [2]. The recycling and reuse of leached GNF in GH irrigation settings allow growers to reduce freshwater and fertilizer needs. Irrigation wastewater reuse has become a well-recognized effective way of plant nutrient reclamation and water conservation pathway [3,4].

Quality water is one of the most important factors to produce healthy GH crops. Impaired water could cause slow growth or even gradual death of a plant. The presence of highly soluble salts and alkalinity interferes with nutrient uptake causing nutrient deficiency and ultimately reduces plant growth. The threshold limits for water elemental components such as potassium (K), calcium (Ca), magnesium (Mg), sodium (Na), chlorine (Cl), and HCO_3 (bicarbonate) are important, as they are essential for plant uptake but also harmful as well if proper concentration limits are not maintained. The high salt concentrations directly injure plant roots, leaves, and stems. For example, potassium concentrations above 234 mg/L caused significant injury to the root of apple seedlings [5]. Salts also can accumulate in plant leaf causing burning of leaf edges, and the pH impact of high alkalinity interferes with the plant nutrient uptake [2]. At a pH above 6.0, it is difficult for plant roots to intake phosphates, Fe, Mn, Zn (Zinc), Cu (copper), and Boron, resulting in deficiency symptoms of yellowing [6].

The recommended pH limit for tomato, bean, and rose is 5.50. A pH higher or lower than this value abnormally lowers plant yields. This impact of pH is sometimes assumed to be pathogenic. Necrotic spotting, streaking, or blotches are characterized as a Mn deficiency but confused with a viral impact. Iron (Fe) or Ca deficiency may also cause yellowing [7]. Disordered growth of young leaves, upward cupping of leaves, and "witches' broom" caused by boron deficiency may be confused with fungal or viral diseases [7]. Excess Cu and boron toxicity caused necrotic leaf spots are confused as fungal impact or sometimes assumed as pesticide damage. Higher than the limit concentration of micronutrients metals (fertilizer) may cause wilt in young plants, which is blamed on a damping-off problem from fungal impact [7]. Acceptable levels for Na and Cl in the case of tomato plants are 0.7 and 0.9 mmol/L, respectively. This limit should not be exceeded. Sodium can quickly become

toxic, as its uptake by plant is very low and accumulates in the rootzone area. Chlorine reduces the uptake of NO_3/PO_4 . When a plant needs to take water, the osmotic pressure of soluble nutrient in water prevents the uptake. Accordingly, the limit of electroconductivity must be maintained for proper growth. For a tomato plant, the limit is 1.6 mS/cm in closed hydroponic circulation and 3.7 mS/cm in the root zone [8].

Plant pathogenic impact is another issue in GH production, and it requires investigation [9]. A literature search revealed that, dissimilar to viruses, some bacteria and fungi such as Ectomycorrhizae are beneficial to plant growth [10]. Major groups of reported plant pathogens are (1) fungal, (2) bacterial, and (3) viral. Member of the first group cause damping off, root and stem rots, cucumber death, and blights of grasses and fruits. They spread from plant to plant by the movement and growth of mycelium, and spores, survive over winter and spread with the movement of diseased plants, soil, and with worker's shoes [11]. These are soilborne pathogens that are not spread through water circulation. They enter the plant through a natural opening such as stomata, wounds, and plant cuticles [12]. The group (2) pathogen is also a soilborne pathogen. It infects via wounds, root tips, or cracks of plants; colonizes the root cortex; invades the xylem vessels; and reaches the stem and aerial parts of the plant through the vascular system [13,14]. This pathogen can easily be spread by footwear, movement of infected plant (rosaceous) materials, and insects [13]. Group (3) members include diversifications and are destructive to tomato, peppers, lettuce, and other crops [15,16]. Impacted plants are dicots, monocots, a wide range of ornamentals, vegetables, and field crops [17–19]. Interestingly, they are transmitted mostly via flies, whitefly vector, grafting, and thrips vector not through water. Additionally, no direct treatment is available for virally infected plants, only to remove the infected plant. Insecticides may be effective in stopping transmission vectors but cannot kill any viruses. The above interpretations lead to the conclusion that, in leached GNF, plant-harmful pathogens may be expected, as water is not the main plant pathogen carrier.

For treatment and recycling of GNF, conventional wastewater and industrial wastewater treatment strategies and methods are not applicable due to restrictions for some treatments when considering plant safety. Treatment by adding hydrogen peroxide, chlorine, chlorine dioxide, and copper ions is shown to cause phytotoxic effects on the circulating nutrient water [20]. Ozone or other oxidizing agents, if present, must be removed from the circulation water to avoid phytotoxic effects in the root zone [21]. Chlorine or other disinfectant has a phytotoxicity effect on the root zone at higher concentrations. Thus, the World Health Organization (WHO) Guidelines suggests a multi-barrier treatment approach for the safe use of irrigation wastewater or GNF in an agricultural setting, so that the reuse of water can allow GH growers to reduce the amount of water and fertilizer [3].

The WHO Guidelines suggested treatments are (i) sedimentation, (ii) flocculation, filtration, and (iii) natural die-off of microbials (if any). Based on the site-specific concerns, GH operations revealed variable water qualities, quantities, and vulnerabilities, requiring site specific treatments and managements [22]. Physical treatment may include filtration, coagulation, and wet pond. The amorphous phase sorption technique in enhanced coagulation could be a promoter in these treatment methods [23,24]. Conventional treatment of wastewater and farm-level irrigation water with slow and rapid rated sand filters were discussed [25]. Different kinds of filters and soil texture to separate different kinds of contaminants have been proposed [26]. Media sorption removal was also reported [27]. Activated carbon sorption was successfully applied in removing contaminants from fruit processing wastewater [24]. Non-activated carbon such as hydrochar is also recommended [3,28]. The above media filtrations reduce a certain % (20–60) of contaminants from the contaminated water. If a tighter membrane, such as RO filtration is used it can reduce about 99% of contaminants including monobasic metals.

Commercially available membranes include ultrafiltration (UF), microfiltration (MF), nanofiltration (NF) and RO, arranged chronologically in decreased pore sizes [29]. RO is widely applied as the leading technology for critical water purification [30]. Production of boiler feed water from wastewater [31], tertiary treatment of wastewater into potable

water [32], separation of fermentation broths [33], concentration of fruit juices [34], removal of Boron and Silica [35], reclamation of wastewater [36], and recycle–reuse of fruits processing wastewater removing 99% contaminants [24] are a few among numerous applications of RO. Even UF and MF membranes are reported in fungi and bacteria removal [37,38]. However, UF and MF are not capable of reducing highly concentrated phytotoxic metal ions. NF can reduce about 50–70%, while RO can reduce about 99% of soluble metals [23]. For recycle of GNF, the partial reduction of micronutrients are required depending on the GNF characteristics. Thus, if a tight membrane such as RO is used, additional fertilizer (nutrients) is required to increase the nutrient level in the permeate. Reject from the RO can be considered.

Based on the review, treatment requirements, and WHO guidelines, the objectives of the research were set to assess different non-chemical multibarrier treatments (a) coagulation filtration (b) hydrochar (HC) and activated carbon (AC) sorption, and (c) RO filtration, along with characterization of GNF collected from GHs. Sorption is the main-stream treatment and sorption agents. The applied HC and AC were produced from GH tomato plant biomass (TPB). RO and a best performing commercial AC were used for performance comparison with the performance of produced HC and AC. The establish treatment approach was evaluated by studying the quality of the leached GNF and measuring the concentrations of the targeted micronutrients. Specifically, could the GNF be safely recycled without impacting the crop plants. Doing so addresses the resource recovery and reuse of waste materials contributing to environmental sustainability.

2. Methods and Materials

2.1. Methods

Optimum coagulant dosage was determined by running individual coagulation in jar tests using the GNF water to be treated. Lowest solubility conditions (pH) of respective coagulants (Al, Ferric chloride) were determined using the gibbsite solid/amorphous solubility diagram. However, Ferric chloride provided an adverse result with GNF treatment, and is not included in results. The details of the treatment by Ferric chloride, alum and PACL coagulations are given in an earlier study Jamal-Uddin [24].

For activated carbon (AC) sorption, tests were completed to obtain the maximum amount of sorption per mass of AC. The optimum dosage of AC and time were determined from analytical results of treated (sorbed) water. The details of the treatment by sorption using AC were given in [24].

RO filtration experiments were conducted using the setup and the procedures described in [24]. For the membrane, Polysulfone-based Filmtec polyamide thin film composite (PA-TFC) extra-low energy (XLE) RO membrane was selected, Model BW30XLE (DOW, Midland, MI) with the nominal flux of 823–1023/8.6 LMH/bar, and MWCO of 100 Da. Operating pressure was maintained at about 125 psi at all times.

2.2. Analyses

For the measurement of pH, electrical conductivity, temperature and TDS, Hitachi Multi Parameter Meter (HACH HQ 40 d Multi-cat. No 58258-00; London, ON) was used. Turbidity was measured with a OAKTON Turbidimeter T-100 (Environmental Express, Charleston, SC, USA). Total nitrogen was measured following the TNT 826 method and using spectrophotometer DR 5000. Simplified Total Kjeldahl Nitrogen (TKN) was measured following the procedure TNT 880 and using spectrophotometer DR5000 by HACH. Chlorine as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) was measured either by spectrophotometer at wavelength 530 nm or by colorimeter at 520 nm using DPD (N,N-diethyl-p-phenylenediamine) as an indicator.

Metal concentrations were measured using DIONEX ICS 2000 Ion Chromatography (Thermo Fisher, Mississauga, ON, Canada) using standard methods.

Pathogens were measured using filtration and agar plating/broth culturing methods to detect the presence of pathogens in GNF. Pathogens were collected on the surface of

0.45- μm and 0.2- μm Millipore filters by filtration, followed by agar plate culture and the subsequent microscopic imaging. The GNFs filter paper was evaluated by optical density (OP) count after 24 h, 48 h, and 72 h of incubations. Microbes of slow or non-responding to culture were confirmed by digitally imaging under a high-resolution microscope. Plate count provided the quantified data, a quantitative tool along with the back-calculation incorporating the dilution factors. For immediate assessment of pathogens Compound Binocular Biological Microscope Model–M827TL (OMAX, Kent, WA, USA) was used.

2.3. Materials

Clear GNF, leached GNF, and GH supply source water (well) were collected with the aid of Ontario Ministry of Agriculture and Rural Affairs (OMAFRA) staff from two local greenhouses (GH1 and GH2) and analyzed and treated immediately upon arrival. It should be noted that the AC and HC used in the present study were produced from the waste tomato plant biomass (TPB) of the GHs from where leached GNFs were collected. Triplicate data were generated in all the cases, and an average result is reported. Both the GHs grow mainly tomatoes, with well water (WW) being the source water. The wastewater conventional sedimentation-filtration followed by nutrients additions is used in clear leach preparation. Leached dirty water from GH2 were collected and analyzed and compared with clear leach of GH1 as no leached dirty GNF was available from GH1. The suppliers of FeCl_3 (ferric chloride), $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$ (Alum), and PACl used as coagulants were given in [24]. Millipore filter papers and analytical chemicals were procured from HACH.

3. Results and Discussions

3.1. Microbial Plant Pathogens Analysis

It is known that viable ‘nonculturable’ organisms such as lower-level plant pathogenic fungi, and oomycetes cannot be detected by the culture method, since they are less specific due to their slow response to culture. Otherwise, almost all the bacterial and fungal pathogens including major part of viruses should be captured by 0.2 μm filtration. Table 1 shows the sizes of pathogen groups, and K^+ (potassium) in comparison with the pore size of the filter. To make the comparison easier, commonly used units were converted to SI units. Table 1 demonstrates that only a part of the viruses may pass through 0.20 μm (200 nm) filters. For easy understanding, if we look to the size of viruses (20–400 nm) in Table 1, it reveals that a 200 nm (0.20 μm) filter can remove part of viruses having size above 200 nm. Any virus size < 200 nm may pass through the 200 nm filter paper. Figure 1. shows 4 plates out of triplicated 27 plates from the three GNFs. It reveals no bacterial presence, and no response to culture was detected even after an extended incubation of 72 h.

Table 1. Size range comparison of bacteria, virus, fungi, and K^+ with RO pores.

Bacteria	Virus	Fungi	Micron Filter	K Ion (K^+)	RO Pore Size
200–1000 nm	20–400 nm	2000–10,000 nm	200 nm	0.15 nm	0.1–1.5 nm
		2–10 μm	0.20 μm	152 pm	1–15 Å
174–871 Da	17–348 Da	1741–8707 Da	174 Da	0.13 Da	0.09–1.3 Da



Figure 1. Cultured plates from filtration (0.2 μm) and media culture experiments (48 h).

To confirm no pathogen response, digital imaging of the surface of the filter was conducted for the raw GNFs. Figure 2a shows microscopic images of the surfaces of 0.20 μm and 0.45 μm filter papers after 48 h of incubation by the magnification of $40\times$. The images in Figure 2a show a very clear surface without any trace of foreign microbial particles or pathogens except the self-fiber networks. Images of some irregular spots, showing the fiber junctions, are found in Figure 2a (bottom). The observed microscopic shapes of three groups of pathogens (fungal, bacterial, and viral) were compared to typical literature view of the three groups as given in Figure 2b [39]. No match was seen, confirming that the tested pathogens were not present in the filter paper of present study. Since there were no microbes in the raw and leached GNF water, the pathogenic assessment of the treated GNF water was considered unnecessary.

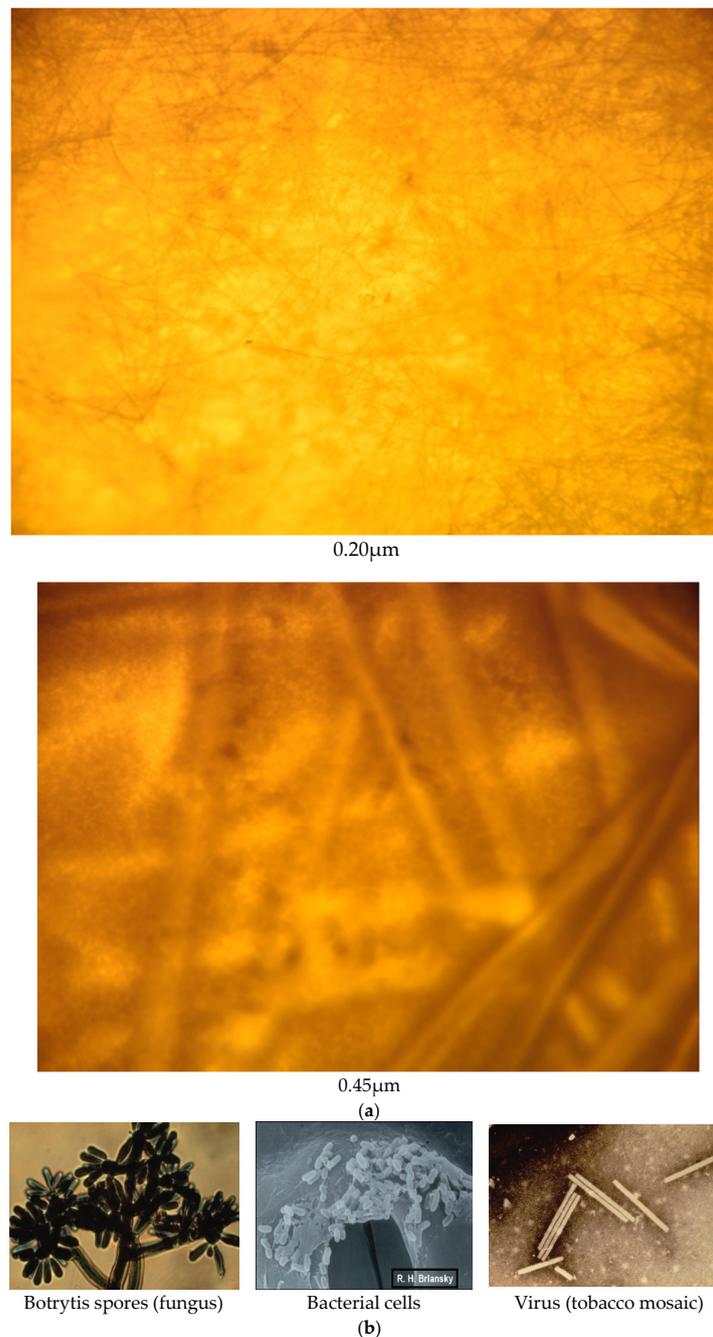


Figure 2. (a) Digital microscopic imaging ($40\times$) of filters after GNF filtration. (b) Microscopic images of the three pathogens.

3.2. Analyses of Leached GNFs including Source Well Water) and Treated GNFs

GNF1 (clear leach) from GH1 was analyzed for pH, conductivity, TDS, and turbidity as an initial step, followed by other required assessments. The GH1 is a tomato greenhouse where well water is used as source water, and after conventional sedimentation-filtration followed by nutrients addition, the water is circulated. GH1 has been facing difficulties in using the well water as the source to prepare clear leach GNF and was looking for the root causes of problems with the water. So, both the source well water and clear leach were assessed. No leached dirty water was collected from GH1.

Treatments of GNF1 were conducted using different coagulants and RO filtration to evaluate their impacts on the quality improvement. Table 2, and b show the results.

Table 2. (a) Results of GNF1 and treated GNF1 water from GH1. (b) Metals analysis results in GNF1 and treated GNF1.

(a)				
	GNF1	Alum coagulation	PACL treatment	RO filtration
pH	7.05	6.63	6.55	6.8
Conductivity ($\mu\text{S}/\text{cm}$)	1262	1300	1440	24
TDS (mg/L)	770	788	798	13
Turbidity (NTU)	2.2	0.24	0.07	Nil
(b)				
Samples Type	Sodium-mg/L (mmol/L)	Potassium-mg/L (mmol/L)	Magnesium-mg/L (mmol/L)	Calcium-mg/L (mmol/L)
Raw GNF1 water	61 (2.65)	670 (17.18)	81 (3.33)	291 (14.55)
CF (11 μ)	25 (1.09)	444 (11.38)	45 (1.85)	107 (5.35)
RO filtrate	1 (0.04)	20 (0.5)	<1 (0.00)	1 (0.05)
Alum treatment				
35 mg/L dosage	21 (0.91)	458 (11.74)	47 (1.93)	109 (5.45)
40 mg/L dosage	21 (0.91)	456 (11.69)	46 (1.89)	106 (5.30)
50 mg/L dosage	22 (0.96)	775 (19.87)	32 (1.32)	57 (2.85)
PACL treatments				
150 mg/L dosage	22 (0.96)	475 (12.18)	48 (1.98)	108 (5.40)
100 mg/L dose	23 (1.00)	644 (16.51)	47 (1.93)	121 (6.05)

CF, cartridge filtration.

The pH of clear GHF1 was 7.05, which was higher than the circulation water pH limit for tomato root zone of 5.5 as suggested in DeKreij et al. [8]. Other limits related to tomato plant are summarized in Table 3. Thus, the GNF1 would be harmful for the tomato plant root zone. Conductivity appears to be in the acceptable range for circulation. Potassium and nitrogen elements are widely applied as fertilizer for plant growth and production. Potassium does not take part in the plant uptake process directly, but it plays a vital role in nitrogen metabolism helping with nitrogen uptake by the plants. Both the deficiency and excess of potassium was found to be a great concern in plant growth and root development. A recent study revealed that both lower than limit and above 460 mg/L (12 mmol/L) of potassium in the circulation water impacts plant growths, including the significant decrease in the growth of the root, leaf, and stem (Xu et al. [5]).

The decrease of nitrate ion flow was also observed, resulting in a lower net photosynthetic rate and photochemical efficiency, which impacted plant productions in a GH setting [5]. The optimum potassium concentration was reported to be 234 mg/L (6 mmol/L) for apple seedlings [5]. Studies on the quality of water and nutrient solution for hydroponic soilless culture of GH suggested different potassium limits for the root zone of the different plants such as tomato 6.5, pepper 5.75, cucumber 6.5, bean 7.0, and rose 2.2 mmol/L, respectively [8]. The potassium concentration in the GNF1 was 670 mg/L which is above the ideal concentration. The calcium concentration in GNF1 was

14.55 mmol/L, which is above the suggested calcium limit of 2.75 mmol/L for a closed system and <8.0 mmol/L for root zone of tomato plant [8]. Therefore, the GNF1 may cause yellowing of tomato plant if it is not treated. Based on the high concentrations of all the phytotoxic metal in the feed WW, simple sedimentation-media filtration would be insufficient to resolve the present issues of reducing metals. Furthermore, with the addition of nutrients, GH1 has to comply with tomato plant root zone limits given in Table 3. Accordingly, GH1 requires a RO filtration of source WW before use or needs to look for new source having better quality water.

Table 3. Tomato plant suggested limits for common nutrients, including conversion factors.

Components	Target for Closed System	Target for Open System	Root Zone Target Values	Unit Conversion Factors mmol/L–mg/L	
EC mS/cm	1.60	2.60	3.70		
pH			5.5		
K ⁺	6.50	9.50	8.00	1	39.1
Ca ⁺⁺	2.75	5.40	<8.00	1	20.1
Na ⁺			<8.00	1	23.0
Mg ⁺	1.00	2.40	4.50	1	24.3
NO ₃ ⁻	10.75	16.00	23.00	1	62.0
Cl ⁻			<12.00	1	35.5
HCO ₃ ⁻			<1.000	1	61.0
H ₂ PO ₄ ⁻	1.25	1.50	1.00	1	91.0
PO ₄ ⁻³				1	26.3

Table 2b gives the concentration of potassium in GNF1 at 670 mg/L (17.18 mmol/L), which was above the suggested 6.5 mmol/L in Table 3. Cartridge filtration (CF) reduced the potassium to 11.38 mmol/L, which was also above the limits, 6.50 and 8 mmol/L for a closed system and for root zone scenario, respectively [8]. Conventional coagulation-filtrations did not reduce potassium or calcium concentrations either, even at the optimized dosages of 40 mg/L and 150 mg/L for alum and PACl treatments, respectively. Hence the simple filtration used was not effective. Although it may reduce turbidity, it seems turbidity is not an issue. High concentrations of dissolved metals that are injurious to plant growth was the issue.

Encouraging results were obtained by RO. The potassium concentration was reduced to 20 mg/L (0.51 mmol/L), which was a reduction of about 97%. The Ca concentration after RO treatment was 0.05 mmol/L, which was a reduction of about 99.4%. As well, RO treatment resulted in the 98.4 and 100% reduction of sodium and magnesium, respectively. Thus, RO treatment can be a suitable solution in resolving all the metallic concentration issues in the source water.

Turbidity was reduced from 2.2 NTU in the raw GNF to 0.24 and 0.07 NTU after alum and PACl coagulation was applied as noted in Table 2. Conductivity increased slightly via coagulation due to the dissolution of added ions from coagulants. On the other hand, RO treatment substantially reduced the conductivity from 1262 µS/cm to 24 µS/cm, and turbidity from 2.2 NTU to nil, respectively, providing a wide scope for readjustment of nutrients concentration for optimal circulation reuse operations. Alternatively, source water having better quality may also be an option for GH1 at this stage to provide well-adjusted targeted concentrations.

The GNF2 collected from GH2 is a leached one after several circulation operations. The analysis shows that it was actually unbalanced with meal ions after plant uptake. Analytical results of leached GNF2 and coagulation products with adjusted pH are tabulated in Table 4a,b. The original pH was 3.77, lower than the suggested value of 5.5. Hence, the pH was adjusted to near 6 to attain optimum effectivity in coagulation experiments. Conductivity of GNF 2 was 2430 µS/cm, about two times higher than untreated GNF1, but below the limit of 3.7 mS/cm suggested for root zone of tomato plant. After pH adjustment, it was 2038 µS/cm. As expected, the TDS follows the trend of conductivity. Similar to GNF1, potassium (15.36 mmol/L) and calcium (12.75 mmol/L) concentrations were above the limits of 8 mmol/L and <8 mmol/L, respectively in GNF2, which may cause yellowing by

calcium and spotted brownish leaf due to lower net photosynthetic rate and photochemical efficiency impacts from high potassium concentration.

Table 4. (a) Analysis of leached and treated GNF2 from GH2. (b) Metals analysis of leached and treated GNF2 from GH2.

(a)								
	Leached GNF2		Alum Coagulation (57 mg/L Dosage)		PACl Treatment (100 mg/L Dosage)		RO Filtrate **	
		Adjusted pH		Adjusted pH				Adjusted pH
pH *	3.77	6.34	4.51	5.90	3.82	3.79	3.49	5.65
Conductivity ($\mu\text{S}/\text{cm}$)	2430	2038	2360	1913	2450	2012	439	71.90
TDS (mg/L)	1334	1260	1281	1048	1324	1151	225	35.70
Turbidity (NTU)	1.40	2.28	0.17	0.42	0.10	0.15	0.10	0.12
(b)								
Samples Type	Sodium mg/L (mmol/L)	Potassium mg/L (mmol/L)	Magnesium mg/L (mmol/L)	Calcium mg/L (mmol/L)				
Leached GNF2	85 (3.70)	599 (15.36)	72 (2.96)	255 (12.75)				
RO filtrate	3 (0.13)	25 (0.64)	4 (0.16)	6 (0.30)				

Notes: * pH of leached GNF2 was low, so coagulation at adjusted pH was conducted for the optimum results. PACl is not pH-dependent. ** RO operating conditions: pressure 125 psi, flow 0.6 gpm.

Similar to the earlier observations for GNF1, coagulation did not reduce conductivity of GNF2, but RO was very effective in reducing the conductivity (Table 4). After pH adjustment, the turbidity of GNF2 was reduced from 2.28 NTU to 0.42 NTU and 0.15 NTU by alum and PACl coagulations, respectively. The high conductivity values of GNF2 indicate the presence of excess nutrients ions in the solution in comparison to GNF1, but they are still below the allowable limit. It should be noted that coagulation did not show any positive effect on the reduction of metal ions in the earlier experiments with GNF1. As such, the metal analysis of coagulation treated GNF2 may not be important at this stage.

The potassium concentration of GNF2 was 15.36 mmol/L, which is slightly lower than raw GNF1 but still above the suggested limit of 8.00 mmol/L. Similar was the case for calcium with 255 mg/L (12.75 mmol/L) of GNF2, which was also lower than GNF1, but above the limit <8 mmol/L. Reduction by RO treatment was 96.50%, 95.80%, 94.50%, and 97.70% for sodium, potassium, magnesium, and calcium, respectively, which were slightly lower than in those of GNF1. The reason of this difference was the lower pH (3.77) of GNF2, which caused acidic injuries to the membrane. However, after pH adjustment the results from fresh RO membrane demonstrated higher metal ion separation than before pH adjustment. It should be noted that RO experiments should not be conducted at a pH lower than 4.50. From the analysis, freshly prepared GNF1 metal concentrations (K, Mg) were higher than leached GNF2, which was imbalanced after several circulation revealing lower quality of GNF1 and source well water.

To cross-check the results in GNF2, the second batch of GNF collected from second greenhouse was marked as GNF3 and subjected to the analysis. Table 5 shows the results. Similar to GNF1 and GNF2, coagulation did not reduce the conductivity of raw GNF3 (2.49 mS/cm), but RO reduced substantially to 0.17 mS/cm (Table 5). The pH of GNF3 (4.34) was slightly higher than the GNF2 (3.77) but still below the root zone limit of 5.50 for tomato plant. The turbidity (0.28 NTU) of GNF3 before the pH adjustment was lower than both GNF1 and GNF2, which was reduced further to 0.12 and 0.002 NTU by alum and RO treatments, respectively. The main cause was the PACl dose being higher for the treatment of GNF3 than the other GNFs, which increased slightly. Turbidity (0.28 NTU) was still very low, so turbidity is not a concern.

Table 5. (a) Baseline analytical results of leached and pretreated GNF3 from GH2. (b) Metal analysis results of GNF3.

(a)				
	GNF3	Alum Dose (59 mg/L)	PACL Dose (200 µL/L)	RO Filtrate
pH	4.34	6.00	3.68	3.88
Conductivity (µS/cm)	2490	2670	2770	170
TDS (mg/L)	1467	1495	1544	89
Turbidity (NTU)	0.28	0.12	0.42	0.002
(b)				
Samples Type	Sodium mg/L (mmol/L)	Potassium mg/L (mmol/L)	Magnesium mg/L (mmol/L)	Calcium mg/L (mmol/L)
GNF3	70 (3.04)	614 (15.74)	182 (7.49)	329 (16.45)
RO filtration	2 (0.09)	17 (0.44)	3 (0.12)	6 (0.30)
Alum treatments				
52 mg/L dosage	101 (4.39)	623 (15.97)	185 (7.61)	336 (16.80)
60 mg/L dosage	102 (4.43)	633 (16.23)	183 (7.53)	341 (17.05)
PACL treatments				
200 mg/L dosage	71 (3.09)	619 (15.87)	187 (7.70)	338 (16.90)
150 mg/L dosage	73 (3.17)	627 (16.08)	189 (7.78)	342 (17.10)

Notes: Leached water pH was adjusted to 6.0 prior to alum coagulation.

The potassium (15.74 mmol/L), calcium (16.45 mmol/L), and magnesium (7.49 mmol/L) concentrations were all above the allowable limits of 8.00 mmol/L, <8.00 mmol/L, and 4.50 mmol/L for potassium, calcium, and magnesium, respectively, for tomato plant root zone (Table 3). Although the potassium concentration was similar to GNF2, the calcium concentration was higher than GNF2. An added issue was that magnesium concentration in raw GHF3 (Table 5b) was much higher than raw GNF2. However, the reduction of metal concentrations by RO filtration was as high as 97.14%, 97.23%, 98.35%, and 98.18% for sodium, potassium, magnesium, and calcium, respectively, which are higher than the case of GNF2 at a normal pH.

The results reconfirmed that both GH2 and GNF3 water qualities (pH, K, Ca, and Mg) are phytotoxic and injurious to the tomato plant root zone. These impacts on plant growth, if not investigated and confirmed, could be blamed on pathogens

3.3. Evaluation of Nutrient Concentrations in all the GNFs

Nutrient levels in GNFs need to be assessed prior to disposal as the impact on lake system eutrophication is dependent on the nutrient (N, P) level of disposed water. Table 6 summarizes all the data obtained by the analysis of untreated GNF1, GNF2, and GNF3 along with those for chlorine and aluminum. Table 3 also includes well water (GH2) analysis and the Provincial Water Quality Objectives (PWQO) which shows the allowable limits for nitrogen and phosphorous nutrients in the Province of Ontario.

Table 6. Nutrient analysis results for all the GNFs and well water from GH1.

Nutrients–mg/L	GNF1	GNF2	GNF3	WW	PWQO
NO ₃ , & NO ₂ -N (mmol/L)	226.00 (3.65)	184.00 (2.97)	319.50 (5.15)	0.50 (0.01)	20 µg/L
TKN	231.50	175.00	165.00	151.00	(Unionized NH ₃)
TN (mmol/L)	457.50 (32.68)	358.50(25.61)	494.50(35.32)	151.50(10.61)	
Phosphate total PO ₄ ⁻³ (mmol/L)	132.00 (4.17)	154.2 (4.87)	315.00 (9.95)	41.9 (1.32)	20 µg/L
Phosphate reactive (mmol/L)	67.00(2.12)	70.50(2.23)	175.00(5.53)	0.50(0.02)	
Free chlorine	0.10	0.20	0.02	0.00	
Chlorine total	0.23	0.22	0.10	0.02	
Aluminum	UMR	Negative	0.051	Negative	
pH	7.05	3.77	4.34	7.75	

Notes: UMR is under measuring range.

The well water in Table 6 is classified as Class 2 category water according to OMAFRA [40]. Table 7 shows the quality of Class 1 water. Based on Ontario regulations disposal limits for GNF nitrogen and phosphorous, Table 7 [41], as well as the PWQO limits in Table 6, make it clear that none of the GNFs are qualified for the safe disposal. RO treatment or land level nutrient management is essential at this stage to prevent eutrophication and to comply with regulations and water quality objectives PWQO and O. Reg. [41].

Table 7. GNF hydroponic source water and disposal quality.

Class 1 Source Water for Hydroponic (OMAFRA)		Leached GNF Safe Land Disposal (O. Reg. 300/14)	
Electric conductivity (EC)	<500 $\mu\text{S}/\text{cm}$	NH_3/NH_4	0.10 mg/L
Na (mg/L)	<30	Nitrite, Nitrate	0.04 mg/L
Cl (mg/L)	<50	TKN	0.05 mg/L
SO_4 (mg/L)	<100	Phosphorous	0.10 mg/L

To establish a comparative scenario and to assess the addition of nutrients (fertilizer) by GHs in the source water and to prepare clear leach (water without any circulation), both WW and leached water were analyzed and compared. Table 8 shows the results for WW, clear leach, and the RO filtrate (permeate). The pH of WW was 7.75 which was reduced to 3.82 at GH2 when the clear leach was prepared to feed the system. Similarly, conductivity was changed from 565 $\mu\text{S}/\text{cm}$ to 2038 $\mu\text{S}/\text{cm}$ after addition of nutrient salts and fertilizer. Due to pretreatment, turbidity was reduced from 11.43 NTU of WW to 0.18 NTU in the clear leach. Hence, turbidity does not cause any concern. Individual metal and nutrient reductions by RO filtration were already interpreted in respective sections.

Table 8. Raw and treated well water results compared with treated and leached GNF2.

	Raw Well Water	RO Filtrate	Leached GNF2	RO Filtrate
pH	7.75	5.00	3.82 (adj. to 6.4)	5.23
Conductivity ($\mu\text{S}/\text{cm}$)	565	37	2038	59
TDS (mg/L)	297	18.34	1201	37
Turbidity (NTU)	11.43	0.06	0.18	0.10
Na-mg/L (mmol/L)	49 (2.13)	2 (0.05)	54 (2.22)	2 (0.05)
K-mg/L (mmol/L)	3 (0.13)	0.00	583 (23.99)	22 (0.55)
Mg-mg/L (mmol/L)	25 (1.09)	4 (0.10)	87 (3.58)	4 (0.10)
Ca-mg/L (mmol/L)	96 (4.17)	5 (0.13)	291 (11.98)	5 (0.13)

Note: Adj. is adjusted.

The GNFs specifications are different for different greenhouses based on their nutrient addition requirements and source water quality. To assess the disposal management, the results of the nutrient analysis given in Table 6 will further be discussed in the following sections.

3.4. Treatments of GNFs by Produced Activated Carbon and Hydrochar

To assess the reduction of micronutrients (metals) and nutrient phosphate (PO_4^{3-}) concentrations, GNF1 and GNF3 were selected and treated by AC and HC produced earlier. Phosphate or orthophosphate is the chemically and biologically reactive component and mostly treated by AC and HC. Two prepared ACs, AC1 and AC2, produced at 700–730 °C for heating durations of 1h (AC1) and 30 min (AC2), respectively, as well as three HCs produced at 260 °C (HC1), and at 225 °C (HC3) were used in this trial. Figure 3a shows the range of K and Ca reduction from GNF1 and GNF3, and Figure 3b shows impacts of excess K and Ca on plant growth. Table 9 shows the results of nutrient (PO_4) removal.

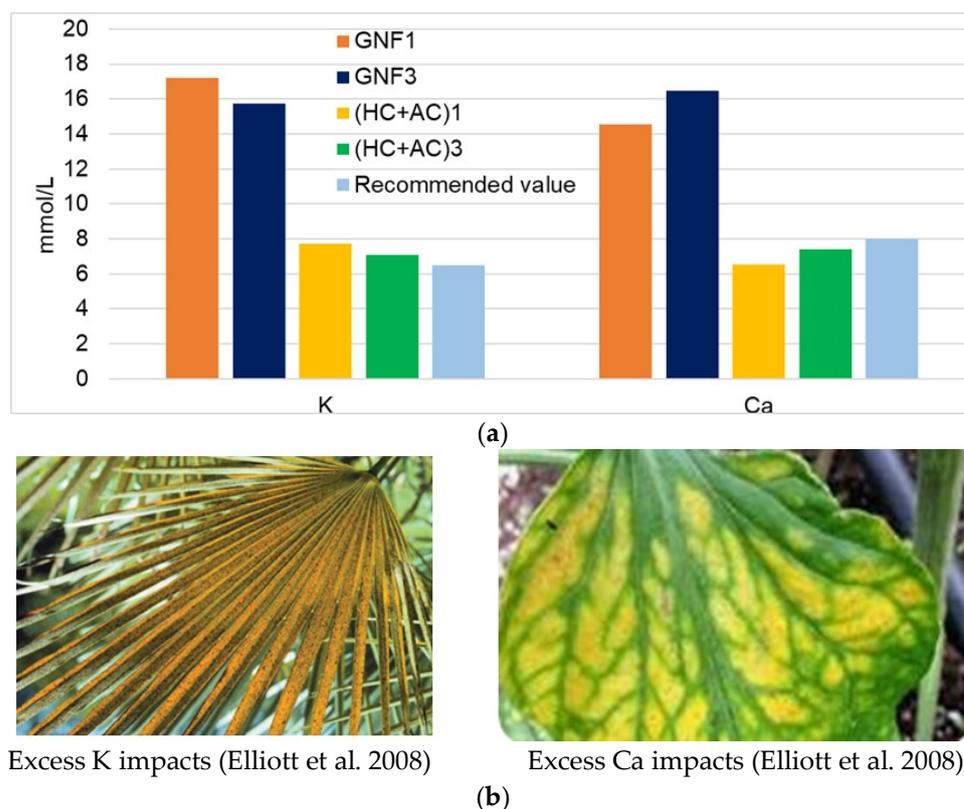


Figure 3. (a) Higher concentrations of Ca and K reduced to a safe recyclable level by HC+AC treatments. (b) Excess K and Ca impacts on plant growth.

Table 9. (a) Reactive phosphate (PO_4^{3-}) mg/L in GNF and treated GNF using HCs and ACs. (b) Results of nutrients removal by AC400 and RO from GNF1 and GNF3.

(a)							
Raw GNF		Treated GNF					
	PO_4^{3-} (mg/L)	RO	AC1	AC2	HC1	HC2	HC3
GNF1	0.61	0.0096	3.3	1.14	1.15	0.78	
GNF3	1.85	0.0221	2.5	0.80	1.95	2.04	3.46
Removal from GNF1		99%					
Removal from GNF3		98%					

(b)				
	Untreated GNF Water (mg/L)		AC400 treated water (mg/L)	
	GNF1	GNF3	GNF1	GNF3
Total nitrogen (TN)	457.00	494.50	188.00	199.80
NO ₃ and NO ₂ -N	226.00	319.50	188.00	188.20
TKN	231.00	165.00	0	11.48
Reactive-ortho PO ₄	67.00	70.50	13.60	63.20
Total phosphate (TP)	132.00	154.20	25.20	125.40
Total nitrogen (TN)% removal			59	60
NO ₃ & NO ₂ - % removal			17	41
TKN% removal			-	93
Ortho PO ₄ % removal			80	64
Total phosphate (TP)% removal			81	19
TKN, TN, Ortho PO ₄ and TP removal% by RO			98–99	97–99

The results of excess micronutrient concentrations reduction by HC and AC revealed that both treatments together may be able to allow circulation, by maintaining the recommended value (RV) given in Figure 3a. The data reveals that after two treatments, the higher concentrations of K and Ca are reduced to be close to the recommended value, allowing for recirculation of the treated GNF.

It may be mentioned here that the phosphate content in both GNFs increased after raw AC and HC treatments, which was attributed to possible residuals of some free phosphorus compounds on the surface of the adsorbents. It was free washed, dried, and reused to obtain the desired results. GNFs were also treated using RO. The results showed a substantial reduction (>98%) of all the nutrients, which could confirm that RO would be the best option if the treatment of GNF should comply with regulations of direct disposal. Considering RO, direct disposal restriction can be achieved easily with economic gain and environmental benefits.

A summary of the nutrients in GNF is shown in Figure 4, revealing higher concentrations in nitrogen components such as TN and NO_3/NO_2 , and orthophosphate is the lowest. Otherwise, orthophosphate is very reactive and interacts with other components involved in environmental transformation and metabolism. For this reason, P is regulated to protect algae blooms in Lake Erie.

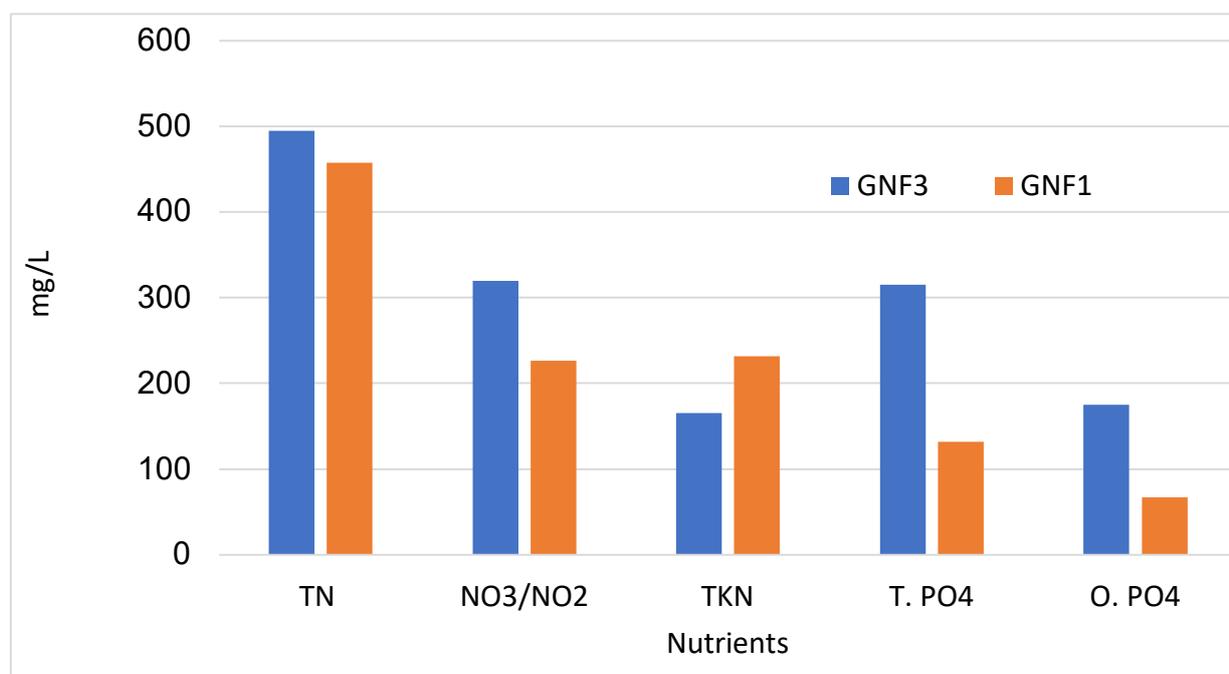


Figure 4. Summary of the nutrients in GNFs.

Earlier evaluations in the phosphate removal experiment showed that the best performing commercial AC was AC400 (M8) [24]. Table 9 shows the results of raw and treated GNF1 and GNF3 when using the best commercial AC400. Results revealed that removal of total nitrogen (azote) from GNF1 and GNF3 were 59% and 60%, respectively. Removal of total phosphate was 81% and 19% and that the amount of orthophosphate from GNF1 and GNF3 was 80% and 64% respectively. The range of nutrients removal by AC400 from the two GNFs was 19 to 81%, while the removal by RO treatment was ranged from 97% to 99% for all nutrients. Therefore, RO filtration is the best treatment to remove nutrients from GNF. However, if RO is used, additional fertilizer will be required to adjust nutrient concentrations in the permeate to prepare clear GNF along with reject management.

Removals of nutrients from two GNFs by different treatments (AC and AC+HC) are comparatively shown in Figure 5a,b. Figure 5a is the removal of nutrients from GNF1 and

5b is the removal from GNF3. Results reveal that TN, NO₃/NO₂, TP, and OP (orthophosphate) are lower in GNF1 (Figure 5a) compared to GNF3 (Figure 5b), but TKN is higher in GNF1 and lower in GNF3. This behavior is expected from clear GNF1 and leached GNF3, respectively.

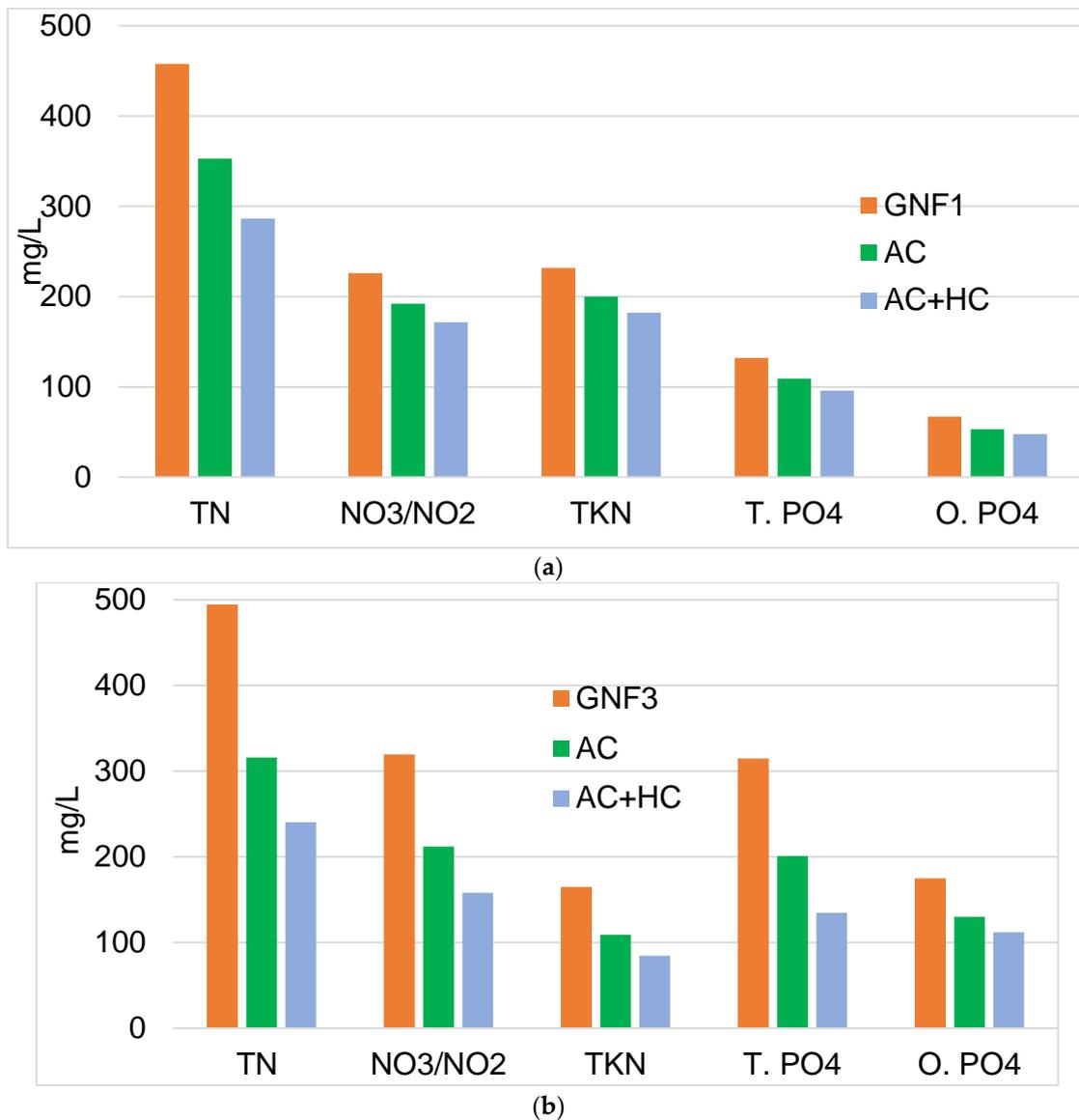


Figure 5. (a) Nutrients in GNF1 and removal by AC and AC+HC. (b) Nutrients in GNF3 and removal by AC and AC+HC.

The GNF disposal options were assessed based on different treatments: HC, AC+HC, and RO. Figure 6 shows a summary of removal by each treatment along with a GNF disposal limit. The dotted red line in Figure 6 shows P+N+K total concentration limit of 140 mg/L for yearly disposal of only 1400 m³/ha. If combined nutrients concentration exceeds this value of 140 mg/L, only a disposal of 700 m³/ha is allowed, which is a great challenge for GHs as they are producing above the limit of 1400 m³/ha. None of the treatments except RO can provide nutrient concentration way below those limits as shown in Figure 6. However, if GNF is filtered by RO, it would be wise not to dispose the pure permeate but reuse as GNF after adding fertilizer. (For clarity, it requires to explain the origin of P+N+K. The P is calculated as TP*2.29, N is calculated as (NH₃-N + NH₄-N +

$\text{NO}_3/\text{NO}_2 + 0.3$) * organic nitrogen (i.e., $\text{TKN}-\text{NH}_3 + \text{NH}_4$), and K is calculated as $\text{TK} \cdot 1.2$). Please note that disposal PNK is not the same as NPK used to standardize fertilizer.

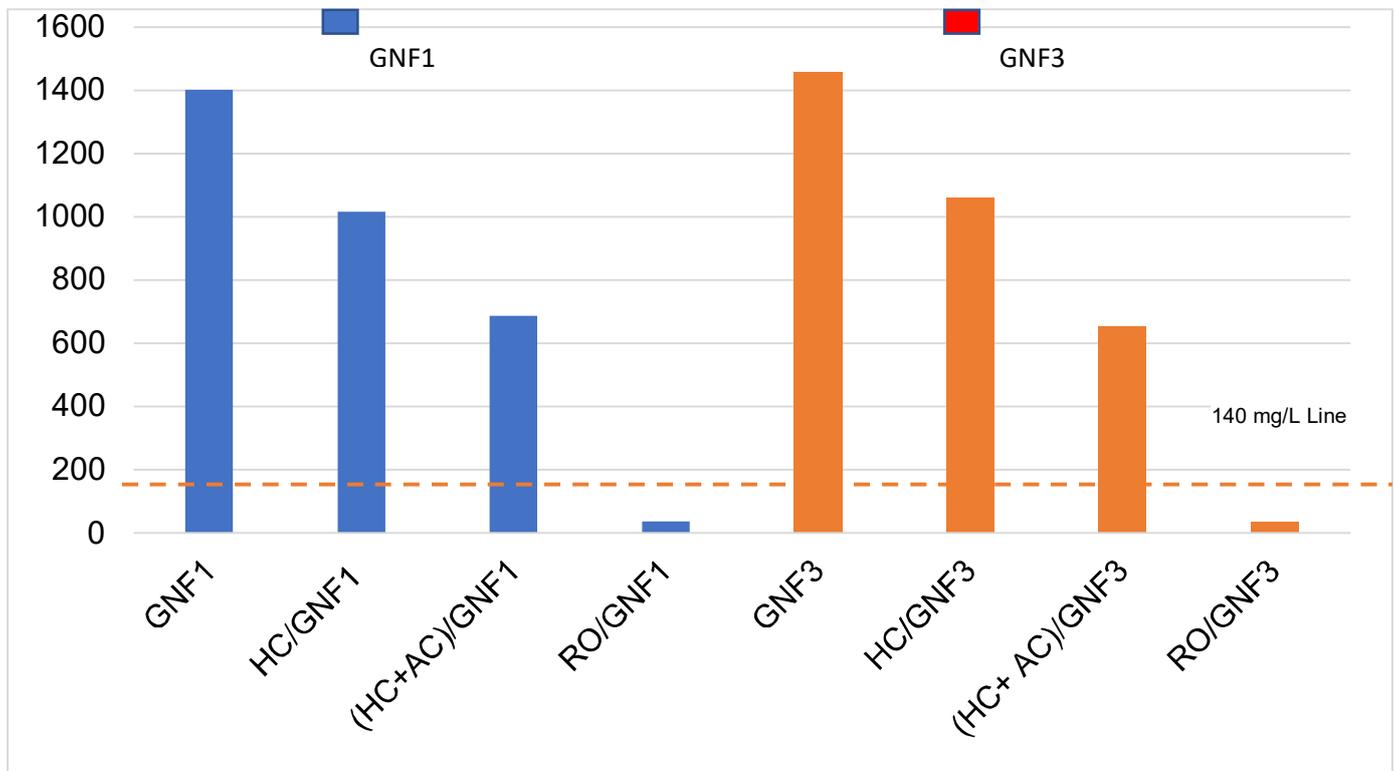


Figure 6. GNF disposal assessment after different treatments.

RO filtration is often considered as an expensive process due to the high-pressure requirement to overcome the osmotic pressure, system loss and liquid head. In contrast to the high salinity of seawater, however, the salinity of GNFs hardly surpasses 2000 mg/L, corresponding to an osmotic pressure of about 1.4 bar (20–22 psi). Therefore, a maximum of 6–8 bar was applied in this research where a bench scale flat sheet system was used. It should be noted that the bench scale unit with flat sheet membrane suffers higher pressure losses compared to the commercial plant with membrane modules. Using a hollow fiber or spiral wound module in place of flat sheet membrane would lower the operating pressure in the actual plant application, making it more cost effective. Moreover, a simple locally available low pressure (6–8 bar) pump can be integrated with an energy recovery device (ERD), which further reduces the energy consumption. Guidelines for the online management of membrane operations for steady performance are also available [24].

Figure 7 illustrates a RO system design for GNF recycle management. An energy model available in the literature would be helpful to calculate energy consumption and to select a required pump size [42]. The presence of any pathogens will be eliminated by using the reject to the hydrothermal process at 220 °C, since pathogens are killed at 95 °C [43,44].

To resolve land disposal issues without treatment, different land-level BMPs such as bio-filters, inorganic filters, constructed wetland, vegetated filter strips, bioretention swales, and bioretention basins were considered to reduce or effectively control the nutrients concentrations in the runoff and comply with discharge limits during crop production using GNF as fertilizer. Simply using a single BMP does not provide an efficiency that meets the discharge standards, suggesting the need for more supportive treatment (or use of multiple BMPs) [40]. To estimate the extent of treatment required to comply with discharge standards while using BMP/s, the most informative SWAT (Soil & Water Assessment tool) model can provide necessary help and GHs may consider using it [45]. About 100% reduction of phosphorous load by SWAT evaluation and applying multiple BMPs was

achieved [46]. The successful use of land-level BMP approach seems to be an alternative to RO treatment for disposal management if run-off quality parameters are not within the regulated limits.

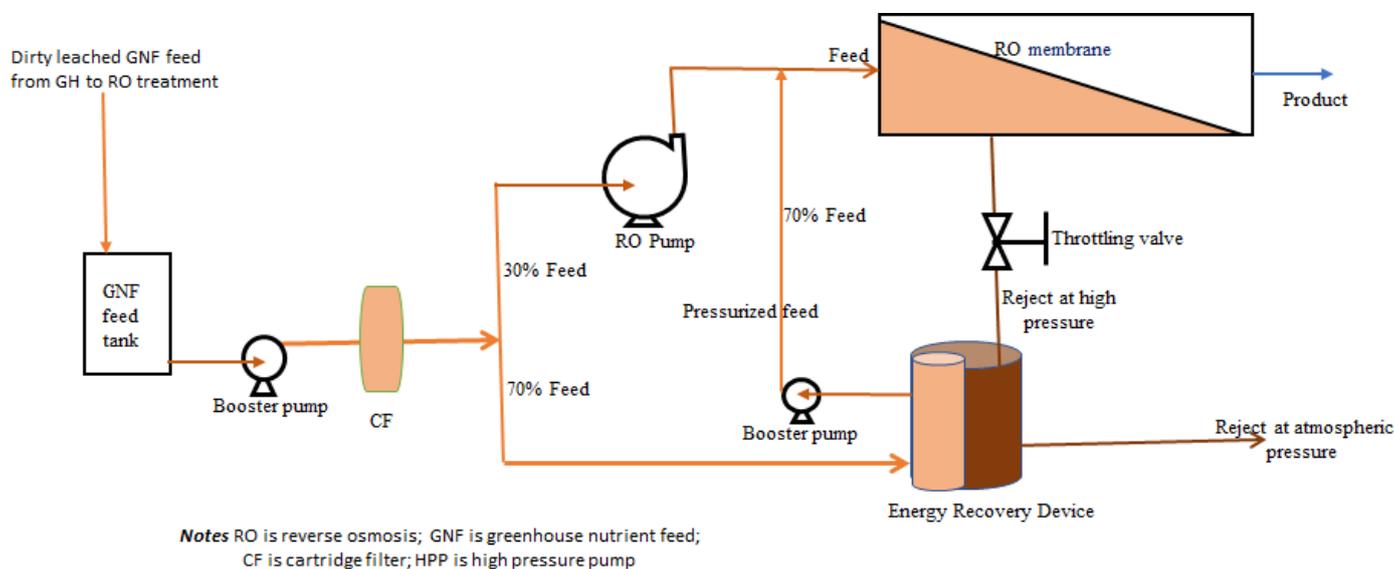


Figure 7. Simplified schematic diagram of an energy economic RO design.

4. Application of Findings

GNF waters were analyzed using different techniques to assess target contaminants. A notion prevails that pathogenic impacts are the major cause of impaired plant growth. Microbial tests for all the GMFs revealed no pathogens in any of the GNFs, which may attribute to the fact that plant pathogens are not waster based. Rather they are soil based whether fungal and bacterial, as well as vector based viral spreading, unlike human pathogens.

The chemical analysis of GNFs revealed higher concentrations of some micronutrients metal ions along with higher pH and conductivities. Even in a freshly prepared GNF from one of the GHs, those constituents were higher than the limit inducing phytotoxicity in the initial GNF before any circulation. The reason identified is the abnormal source water specification, suggesting the requirement of alternative source water or pre-treatment prior to addition of fertilizer. Analyses of leached GNF revealed imbalanced metal concentrations for potassium (15.75 mmol/L), calcium (16.45 mmol/L) and magnesium (7.43 mmol/L), which were above the allowable limits for tomato plant root zone of 8.0 mmol/L for Ca ad K, and that for Mg of 4.5 mmol/L, respectively.

The safe recommended pH limit for tomato plant root zone is 5.5 and that for conductivity is 3.7 mS/cm, respectively. The measured pH was 4.34 in GNF3, which is acidic and can impact root zone conditions. All the analyses revealed there exist phytotoxicity from constituents in the GNFs that need to be readjusted prior to any recirculation. The impacts of those phytotoxic water conditions are generally blamed on the possible presence of pathogens. Some examples of excess metal ion impacts are presented earlier in the manuscript.

To adjust micronutrient concentrations, different treatments including conventional coagulation-filtration, HC and AC sorption, and RO filtration were applied. None of the treatment of coagulation filtration, and sorption could solve the issue when used stand alone. However, when performances of HC and AC are combined together, it could reduce the higher concentrations of K, Ca, and Mg to the safe limit as demonstrated in figures, which is encouraging.

The application of RO filtration substantially reduced almost all the nutrients and micronutrients in the range of 98–99% producing very clear water. Complete reduction

is not demanded for recirculation, as it consumes additional fertilizer to adjust the NPK concentrations to prepare a fresh GNF. However, this is used as an option if a GH requires such treatment they may use RO filtration. In addition, when a GH is required to reduce some of their GNF water by land disposal to comply with regulations, RO filtration provides substantial reduction of lake eutrophication nutrients (N, P). It must be stressed that it is not economical to dispose RO filtrate. Other constituents of GNF such as nitrogen, phosphorous, chloride, and aluminum were within the limits of root zone for tomato plant. It was suggested that analysis and desired fertilizer adjustment should be conducted continuously to ensure steady plant growth in GHs.

The results were encouraging for the use of self-produced treatment agents such as HC and AC from waste biomass of tomato plant into treatment GNF and allow recirculation. The research reveals that this is a comprehensive solution option as both the solid biomass and the liquid GNF can be recycled in an environmentally sustainable manner.

Novelty

“The Resource Recovery and Reuse (RRR)” is a global program approach for the safe reuse of agro-industrial wastes. The approach presented was designed to recover resources from two wastes representative from greenhouses (GHs) in Southern Ontario. Nutrient discharges in the project area (Southern Ontario) are strictly regulated to control Lake Erie algae bloom, a binational concern with the highest priority. In this respect, greenhouse (GH) producers in the study area have dual problems of waste biomass and GH nutrient feed water (GNF) management; as land disposals are regulated and are not allowed without proper treatment. GNF is a liquid fertilizer containing plant-required nutrients that need to be recovered. The disposal of waste biomass spreads plant pathogens, while burning causes greenhouse gas emissions. To address both the issues, complete recycle and reuse of (1) tomato plant biomass (TPB) and (2) GNF were the main focus of the research. The TPB was converted into hydrochar (HC) and activated carbon (AC), which were used as treatment agents in the treatment and recycle of GNF without discharge. This approach supported Ontario’s initiatives on nutrient management in the area. Thus, the value of this research for GH business development and environmental sustainability is fairly significant.

5. Conclusions

Higher concentrations of potassium, calcium, and magnesium micronutrients were observed, along with high pH in the leached GNF, which induced phytotoxicity to plants. To resolve the issue obtaining recyclability of GNF following the limits for circulation, different treatments, including conventional coagulation–filtration, hydrochar, and activated carbon sorption, and RO filtration were applied. The hydrochar and activated carbon reduced metal concentrations as required, providing recyclability to the GNF. RO filtration provided substantial reduction of eutrophication nutrients (N and P) to comply with direct disposal limits but advised to reuse the pure permeate water in preparation of clear GNF by adding new fertilizer. It is suggested that analysis and desired fertilizers adjustment should be conducted continuously to ensure steady plant growth in GHs. No pathogens were detected in any of the evaluated GNF waters.

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Abbreviations and Terms:

AC	Activated carbon
CF	Cartridge filter
Clear leach	Freshly prepared GNF
GH	Greenhouse
GH1	1st GH
GH2	2nd GH
GNF	Greenhouse nutrient feed
GNF1	Clear leach from GH1
GNF2	Leached dirty GNF from GH2
GNF3	Leached dirty GNF from GH2
HC	Hydrochar
HTC	Hydrothermal carbonization
Leached GNF	Circulated nutrients unbalanced dirty GNF
LMH	Liters per square meter hour
Micronutrients	Metallic nutrients (salts)
OMAFRA	Ontario Ministry of Agriculture, Food and Rural Affairs
PACL	Poly aluminum chloride
PWQA	Provincial Water Quality Objectives
RO	Reverse osmosis
TGNF	Treated GNF
TPB	Tomato plant biomass
WW	Well water

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