Groundwater nanofiltration process efficiency improvement with additional concentrate membrane treatment

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Abstract

Nanofiltration (NF) of waste water originated from nanofiltration drinking water plant stationed in town of Kikinda (Northern Serbia) was investigated. Experiments on removal characteristics of nanofiltration membranes when exposed to influent rich in arsenic and natural organic matter under different flux and transmembrane pressure (TMP) conditions were conducted in order to obtain drinking water from waste water and reduce total amount of waste water. Applied NF membranes showed remarkable removal characteristics. Also, obtained result for concentrate yield, an indicator of reduced concentrate amount, of 8.89% under optimum flux value presents considerable amount of reclaimed drinking water. Calculated empirical and theoretical concentration factors quotients were indicated to probable accumulation of some inlet water components at NF membrane surface. Based on the obtained results that nanofiltration membranes remove natural organic matter, arsenic, ammonia and sodium with high efficiencies of around 98, 96, 80 and 93%, respectively, the conceptual design of drinking water plant for City of Kikinda was defined and presented.

Keywords: nanofiltration, water reclamation, concentrate yield, arsenic, humic matter, concentration factor.

Membrane processes are currently considered to be one of the best separation technologies for water and wastewater treatment. Nanofiltration has been a rapidly expanding field leading to significant innovations in the last decade especially for aqueous effluents treatment in various industries. About 75% of the total market share of NF membranes worldwide is dominated by water demineralization and wastewater treatment applications [1]. NF is widely used for production of clean and pollutants free drinking water around the world [2–4]. Regardless, NF concentrate disposal presents a great problem because of its quality and quantity [5]. Even though the properties of NF membranes are improving every day in means of materials, resistance and recovery rates [6–8], the matter of concentrate discharge is still present.

Reclamation of membrane processes concentrates separated during desalination of sea or fresh water presented a big challenge in many previous investigations [9–11]. There are also numerous studies dedicated to water reclamation from waste water plants [12–14] in order to reuse obtained product for instance for irrigation [15].

In the Northern part of Serbia – Vojvodina, where the investigations were conducted, the main source of drinking water is unprocessed groundwater. The problem is that the quality of groundwater is not suitable for human consumption, according to local regulations [16]. More correctly speaking, water from approximately 45% public wells has elevated levels of arsenic. Second problem of Vojvodina's groundwater is natural organic matter (NOM) which concentration exceeds maximum tolerable level (MTL) [16] in majority of groundwater used for human consumption. There were many previous investigations based on application of adsorption and other processes for removal of groundwater arsenic and NOM. Strong base macroporous ion exchange resin was successfully applied for adsorption of As and NOM from chlorinated and non-chlorinated groundwater [17]. Since NOM is precursor of disinfection by products formation, adsorption of organics from the groundwater onto resin significantly decreases the possibilities of trihalomethanes arising [18]. The role of individual NOM fractions on changes in trihalomethanes and haloacetic acids formation during coagulation with iron chloride and a combination of polyaluminium chloride and iron chloride was determined. Results showed that most of the dissolved organic carbon (68%) in the raw water comes from the fulvic acid fraction, yielding 41% of the total trichalomethane precursors and 21% of the total haloacetic acid precursors [19]. Laboratory scale electrocoagulation/flotation reactor was successfully applied for removal of high concentrations of natural organic matter and arsenic from groundwater [20]. Based on the obtained results,

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conceptual design of drinking water plant for City of Kikinda was presented.

The electrocoagulation/flocculation reactor at the optimum operational conditions was able to remove 93% of arsenic and 70% of dissolved organic carbon, comparing with raw groundwater [19].

Arsenic can be removed from water in greater percentage when it is present in organic form. It is found in literature that As, if present in natural organic matter-rich water, has ability to make complex compounds with anions of NOM as ligands [21]. Yu and a group of authors investigated effects of ion concentration and natural organic matter on arsenic(V) removal by nanofiltration under different transmembrane pressures using four different types of NF membranes [22]. It is shown that the removal efficiency of arsenic was influenced by ion concentration, TMP, and the presence of NOM.

Therefore, topic that interested authors was behavior of NF membranes when exposed to influent containing high levels of arsenic and humic matter. Aim of this work was to obtain drinking water from the NF concentrate, to investigate influence of transmembrane pressure on NF membranes functionality as well as to study influence of water flux on NF concentrate yield.

MATERIALS AND METHODS

Description of experiment location

Experiments were conducted in northern Serbia (Vojvodina) in the town of Kikinda (45.84° North, 20.45° East). The water-bearing formations in Vojvodina were formed during the final phase of existence of the Pannonian Sea [23]. In this region, the arsenic rich waters have a characteristic yellow color, a specific taste, and increased arsenic concentrations range from 0.040 to 0.380 mg/L [24–26] as well as elevated NH_4-N (ammonia – AM) and sodium level. The yellow color is due to the presence of undesirable organic (humic) matter. Measured physicochemical parameters in groundwater exceeds MTL [16] as presented: NOM, expressed as $KMnO_4$ consumption (*COD*, mg $KMnO_4$/L): 28.65; As [μg/L]: 18.07; Na [mg/L]: 206.90; NH_4-N [mg/L]: 2.07. This water is without any treatment being used as a source of drinking water in majority of nearby settlements.

Design of the pilot plant and operating conditions

Nanofiltration concentrate was obtained from the industrial NF plant, used for production of drinking water from groundwater according to local regulations [16]. NF plant operated under water flux (J = 25.76 L/(h m^2)), and contained nanofiltration membranes type CSM NE8040-90 manufactured by Torey Chemical Korea Inc. [27]. Inlet groundwater flow rate in the first step of nanofiltration was 13,500 L/h. The actual permeate and concentrate flow rates produced in NF plant were 11,500 and 2,000 L/h, respectively. Industrial NF plant permeate (NFP1) physicochemical composition is in accordance with the local regulations for drinking water [16] and it is used as a drinking water on investigated location. For this investigations NF plant concentrate (NFC1), which is usually discharged into the sewage, was treated on a self-made NF pilot plant (NFPP) at different flux conditions. Scheme of NFPP is given in Figure 1. NFPP has been designed for optimal permeate flow rate of 700 L/h, and contained three NF membranes (CSM NE4040-90, Torey Chemical Korea Inc. [28]) in two-array operating regime. As can be seen from the Figure 1, a part of the concentrate produced on NFPP (NFC2) was recirculated and joined with NFC1 – making feed water flow in order to reduce wastewater quantity. Because of this, values of *COD*, As, Na and AM in NFC1 were proportionality calculated with respect to inlet and recirculation flow rates. Experiments were conducted while the hydraulic conditions of the NFPP were operable. After first experimental procedure membranes were chemically cleaned using Clean-in-place method. Cleaning lasted for 2 h and it was done using 0.1% solution of NaOH. At the end of cleaning process solution was dark yellow color with foam appearance on the solution surface. Experimental procedure was repeated two more times. All presented results of groundwater and effluents are the mean values of measured parameters from three experimental procedures.

Physicochemical methods and analytical instruments used in the water analyses

General water quality parameters for the groundwater and effluent, such as *COD*, and arsenic, were determined by standard analytical methods [29]. Total arsenic and sodium was determined by atomic absorption spectrophotometry on a Varian AA 240FS. The total arsenic as well as sodium content was determined by the VGA technique on a Varian/VGA-77. Ammonium nitrogen content was determined by standard spectrophotometric Nessler method on instrument Hanna C-100. The TOC was measured on an Analytik Jena/ /Multi N/C 2100 instrument.

Calculation of experimental parameters

Operational parameters including inlet flow, permeate flow and concentrate flow were monitored during the study. Pressures between the feed and permeate side were detected by a pressure meter. According to the pressures, a transmembrane pressure that presents a force needed for filtration process to occur can be calculated using Eq. (1), where p_f, p_c, and p_p are influent, concentrate and permeate pressure, respectively.

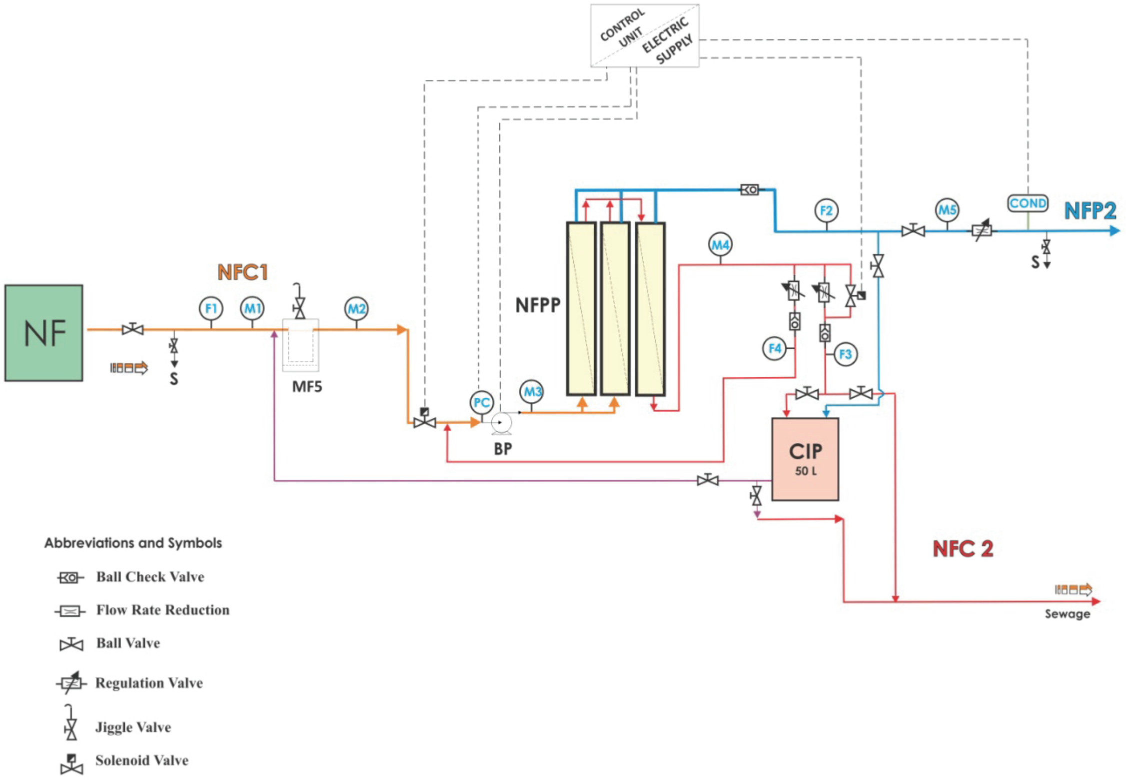

Figure 1. Scheme of the NF pilot plant (NF – industrial NF plant; NFC1 – concentrate after first step of nanofiltration; MF5 – microfilter of 5 mm; BP – booster pump; NFPP– NF pilot plant; CIP– clean in place unit; M1–5 – pressure gauge; PC – automatic pressure unit; F1–4 – continuous flowmeter; COND – conductivity meter; S – sampling tap; NFC1 – permeate after second step of nanofiltration; NFC2 – concentrate after second step of nanofiltration).

Calculation of membrane flux J (L/hm^2) is given in Eq. (2), where Q_p presents permeate flow (L/h), and A presents effective membrane area (m^2):

$$TMP = \frac{p_f + p_c}{2} - p_p \qquad (1)$$

$$J = \frac{Q_p}{A} \qquad (2)$$

Flow rate of permeate produced per flow rate of feed water presents membrane recovery that is calculated as follows:

$$R = 100\frac{Q_p}{Q_f} \qquad (3)$$

where: Q_f – feed water flow (L/h).

Removal efficiency (η) can be expressed using Eq. (4), where c_p and c_f are concentrations of measured component in permeate and feed, respectively, in mg/L:

$$\eta = 100(1 - \frac{c_p}{c_f}) \qquad (4)$$

Concentration of measured parameters in the concentrate, c_c (mg/L), can be calculated using expression (5), where Q_c is volumetric flow (L/h) of concentrate stream [30]:

$$c_c = \frac{(Q_f c_f) - (Q_p c_p)}{Q_c} \qquad (5)$$

The concentration factor (*CF*, the ratio of the concentration of a component in concentrate and feed) can be calculated from the mass balance for each measured water component [30]:

$$CF = \frac{c_c}{c_f} = \frac{Q_f}{Q_c}\left[1 - R\frac{c_p}{c_f}\right] \qquad (6)$$

Concentrate yield, which was calculated using expression (7), is used to show percentage of feed flow rate that is converted to concentrate stream:

$$Y_c = 100\frac{Q_c}{Q_f} \qquad (7)$$

RESULTS AND DISCUSSION

Concentrate nanofiltration was very effective while COD and arsenic concentration in the permeate produced on the NFPP were below the maximum tolerable values [16]. Influence of the transmembrane pressure on the relative removal of measured parameters in NFC2 is shown on Figure 2. As can be seen, removal of all measured parameters with the increase of TMP was progressing in a constant way, with slight decrease of c/c_f values with TMP increase (where c and c_f present concentration of measured parameters in feed and NFC2, respectively). Also, values of c/c_f for *COD*, *TOC*, and As below 0.5 indicate that the experimental membranes showed excellent removal characteristics. Mean values of As, *TOC* and *COD* in P were 2.6 μg/L, 0.33 mg/L, and 1.7 mg $KMnO_4$/L, respectively, while average values of Na is 56.66 mg/L and ammonia were 1.14 mg/L. It can be concluded from obtained results that experimental NF membranes showed excellent arsenic and *COD* removal characteristics which is proven by the fact that concentrations of all measured parameters are below the MTL, except the ammonia value, which slightly exceeds MTL, according to local regulations [16].

Influence of flux values on the membrane rejection is shown in Figure 3. Increase in ammonia removal efficiency was observed with the flux increase, while the mean value of membrane rejection ($\overline{\eta}$) for ammonia was 80.40%. Membranes exhibited similar constant behavior in regard to *COD*, *TOC*, As and Na removal, with $\overline{\eta}$ of 98.23, 98.73, 96.74 and 93.45%, respectively.

$Y_{c,total}$ value (total percentage of produced concentrate after both steps) as well as R_{total} (total recovery after both steps) are given in Figure 4. Concentrate yield after the first step of the nanofiltration was 18.52% and the recovery valued 81.48%. $Y_{c,total}$ values had decreasing trend with TMP increase. Also total recovery increase was observed. Optimum transmem-

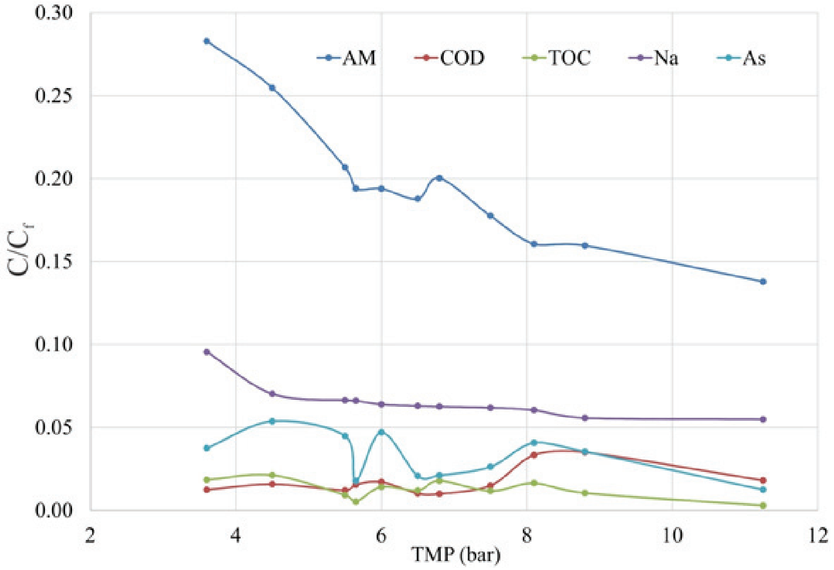

Figure 2. Influence of TMP on the removal of investigated physicochemical parameters in the permeate obtained from NFC2.

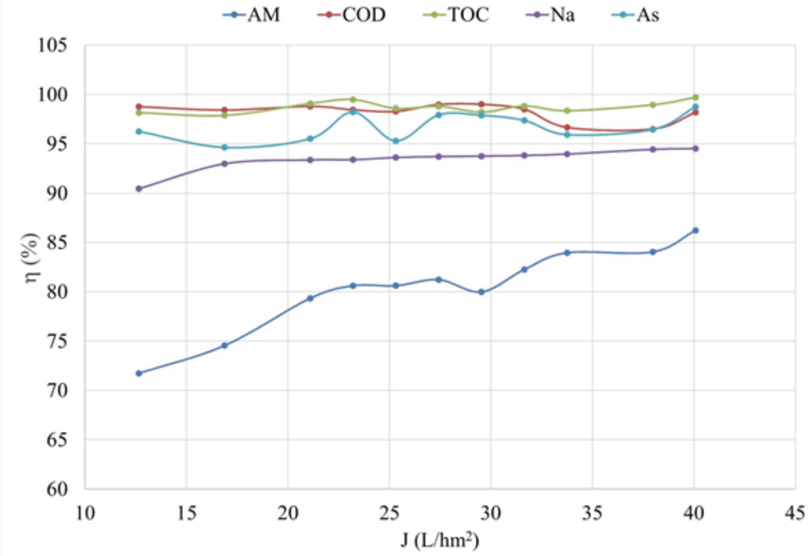

Figure 3. Changes in measured parameters removal efficiency with flux values increase.

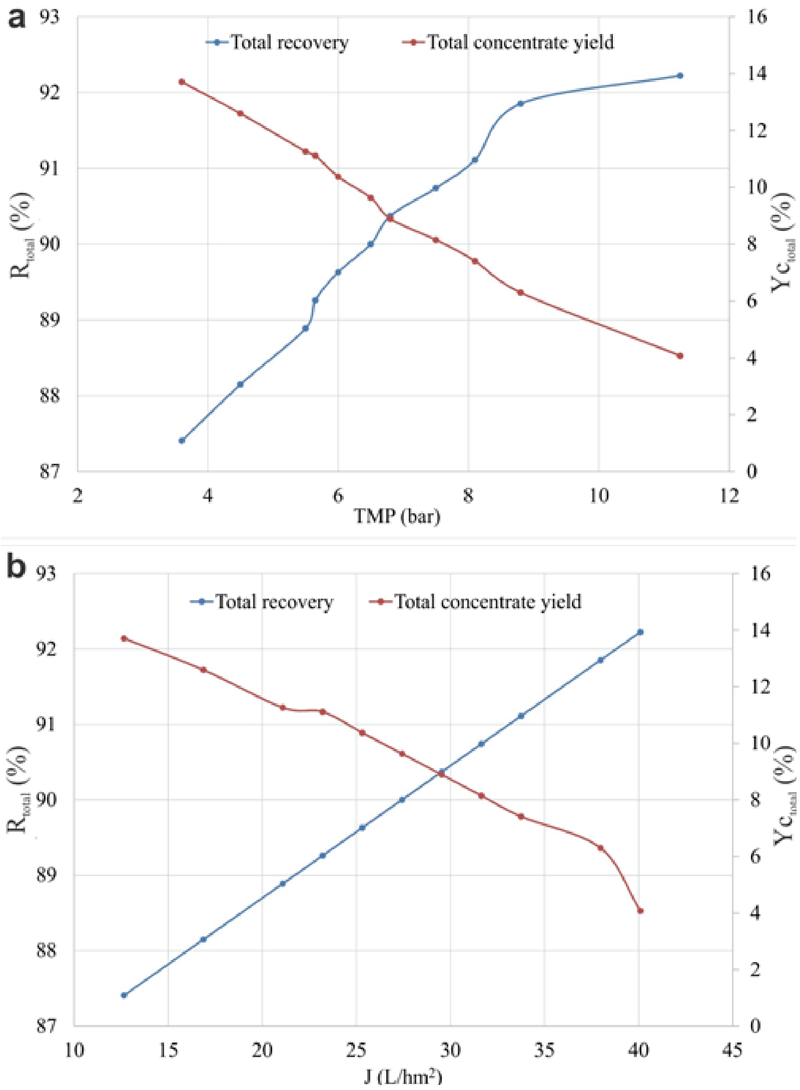

Figure 4. Transmembrane pressure (a) and flux (b) influence on total concentrate yield and total recovery values.

Table 1. Theoretically obtained and empirically measured concentration factors at optimal flux value

Parameter	*AM*	*COD*	*TOC*	Na	As
CF_{th}	2.05	2.16	2.16	2.13	2.15
CF_{ex}	2.41	2.50	1.79	2.55	2.36

brane pressure and membrane flux were between 6.5––7.5 bar and ~30 L/hm^2, respectively, which was in accordance with observed intercepts in Fig. 4a and b. Obtained R_{total} was 90.37% and $Y_{c,total}$ was 8.89% which is significantly lower than presented in previous results [30].

Equation (5) was used to calculate theoretical values of parameters in the concentrate. Concentration factor (expression (6)) was calculated for theoretically obtained results (CF_{th}) and empirically measured values in the concentrate (CF_{ex}). Both concentration factors for different parameters at optimal flux value are given in Table 1. The best and the worst CF_{ex} *vs*. CF_{th} relative accordance of around 9 and 20% were evident in the case of arsenic and *TOC*, respectively.

CF_{ex}/CF_{th} quotient was established as real concentration factor CF_r and it defined passage of some water constituents through, *i.e.*, their accumulation on the membrane surface. CF_r values are presented in Table 2 and showed that all values greater than 1 ensigned good passage of inlet water constituents to the concentrate without or small accumulation on the membrane surface. For fluxes greater than 31 L/hm^2 all CF_r values were less than 1. Exceptions were data for *TOC* which for all flux amounts valued lesser than 1, what denoted to probably high content of chemically non-easily oxidable organics. The similar values less and close to 1 were obtained for arsenic which ensigns the possibility of predominant organically bonded arsenic species. CF_r values for ammonia, easy oxidable organics as *COD* and sodium were in flux range until 31 L/hm^2 greater than 1, and did not show any affinity to accumulate onto membrane surface. Higher fluxes contributed to CF_r decrease below 1, when all determined parameters could potentially be accumulated in the membrane pores.

Table 2. Measured parameters CF_r quotient changes in the dependence of flux

J / L h^{-1} m^{-2}	*AM*	*COD*	*TOC*	Na	As
12.66	1.31	1.47	0.75	1.12	0.78
16.88	1.32	1.30	0.88	1.48	0.64
21.10	1.40	1.42	0.75	1.40	0.61
23.21	1.40	1.33	0.86	1.41	1.34
25.32	1.34	1.24	0.85	1.36	0.58
27.43	1.22	1.23	0.80	1.27	1.15
29.54	1.18	1.16	0.83	1.20	1.09
31.65	1.18	0.91	0.78	1.11	1.04
33.76	1.09	0.94	0.74	1.05	1.09
37.97	0.97	0.84	0.72	0.92	0.88
40.08	0.67	0.67	0.54	0.63	0.68

As mentioned, membranes were chemically cleaned, and *COD*, *TOC*, As, Na and ammonia concentrations were not of interest for this work and were not measured in cleaning solution. Yet, appearance of yellow-brown color established by visual method confirmed data presented in Table 2, that part of humic matter along with other measured parameters were accumulated on the surface of the membrane.

Conceptual design of drinking water plant for City of Kikinda

Described two step nanofiltration of groundwater and further concentrate nanofiltration results were the base for preliminary design of potable water plant (PWP) assigned to supply of City of Kikinda citizens. Input data for dimensioning of PWP capacity are presented in Table 3.

Data presented in Table 3, accompanied with described nanofiltration results were the base for preliminary design of PWP presented in Tables 4 and 5.

Groundwaters under pressure of well pumps pass through double fineness microfilter protective cartridges of 20 and 5 μm to NF booster pumps which increase the pressure to 8 bar. First step nanofiltration process consists of two parallel interconnected NF plants. Obtained permeate, NFP1, flows to the collecting tank of 3,000 m^3 volume by previous disinfection with sodium hypochlorite. Separated concentrate NFC1 flows to additional filtration process into the third NF plant. Secondly originated permeate, NFP2, join the NFP1 permeate stream, and NFC2 as a total concentrate is disposed to the recipient. Complete two step NF process does not required any chemicals in service regime, but only for temporary membranes chemical cleaning.

The PWP nanofiltration equipment installing space required small area of ~300 m^2, plus reservoir needed area of ~1,300 m^2, sized to ~25 m×50 m×4 m as a width, length and height, respectively. The pure drinking water reserve of ~3,000 m^3 is enough for 12 h

Table 3. Essential input data for preliminary design of PWP

Population (people)	Daily consumption L/capita	Water demand m^3/d	Average hourly consumption m^3/h; L/s	Maximal hourly consumption m^3/h; L/s
37,676	150	5,651.40	235.46; 65.41	353.21; 98.12

Table 4. PWP water balance

Feed water L/s	NFP1 L/s	NFC1 L/s	NFP2 L/s	Total permeate L/s	Total concentrate L/s
110	89.65	20.35	9.76	99.41	10.59

Table 5. Technical and economic overview of PWP construction and maintenance

Position	First NF step	Second NF step
Installed electric power, kW	140	35
Number of NE-8040 membranes	288	72
Number of pressure membrane vessels	48	12
Specific electric power consumption, kWh/m^3	0.321	–
Building required, m^2	~300	–
Net capacity, m^3/d	7,064.20	–
Net capacity, m^3/y	2,578,433	–
Capital investment, €	~2.2 million	–
Price of drinking water *vs*. maintenance costs, €/m^3	~0.1	–

continual supplying of citizens, as well as for 8 h of maximal hourly consumption demand. The graphical abstract of the conceptual design which presents 3D view of nanofiltration plant as a part of PWP is shown in Figure 5.

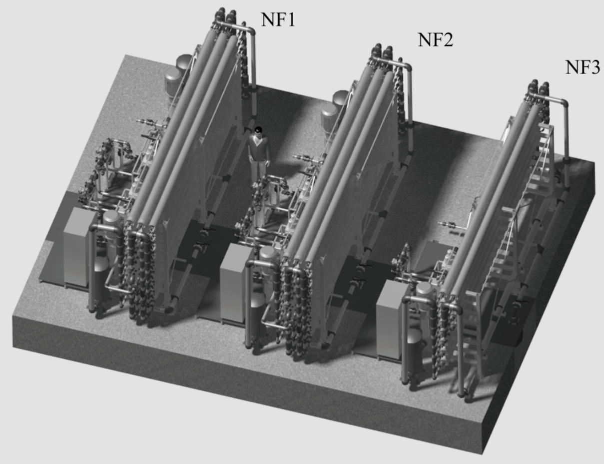

Figure 5. 3D view of NF drinking water plant.

The Figure 5 presents two first step nanofiltration units (NF1 and NF2) for groundwater purification and the third unit (NF3) designed for NFC1 treatment. In comparison of plant dimensions with the displayed worker average height, it could be seen that the main part of PWP does not required big space.

CONCLUSION

Pilot plant used in this study showed great ability to reduce amount of disposable NF concentrate. Arsenic and *COD* values in permeate from second step of the experiment were below *MTL*, according to local regulation and could be mixed with the first step permeate to increase total drinking water yield for almost 9%.

Concentrate nanofiltration exhibited high rejection of all determined water parameters, *i.e.*, ammonia more than 80%, and *COD*, *TOC*, Na and As for over 90%.

Results showed that obviously high organic matter and arsenic amounts in the same solution can be successfully removed by nanofiltration force. At the base of these data investigated concentrate nanofiltration was in the same time wastewater treatment and potable water preparation by mean of physical process without usage of any chemical. Established CF_r quotient is excellent indicator for degree of potential accumulation of water constituents on membrane surface during desalination. Analysis of obtained CF_r values for all investigated water constituents proved that only organic matter coupled with arsenic species definitely was accumulated in membrane pores which could evoke potential fouling.

Obtained results are obviously important contribution to modern trends of preserving natural resources of fresh water. Taking into consideration amount of reduced concentrate, authors suggest application of obtained desalination procedure and results to a large scale membrane plants for drinking water conditioning. Based on the obtained results, conceptual design of drinking water plant for City of Kikinda was presented.

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IZVOD

POVEĆANJE EFIKASNOSTI NANOFILTRACIONOG PROCESA PRERADE PODZEMNE VODE DODATNIM MEMBRANSKIM TRETMANOM KONCENTRATA

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(Naučni rad)

U radu je prikazan tretman otpadne vode (koncentrata) iz nanofiltracionog uređaja za proizvodnju pijaće vode kapaciteta proizvodnje permeata od 11.500 L/h. U cilju dobijanja pijaće vode iz koncentrata kao i smanjenja količine otpadne vode izrađen je nanofiltracioni pilot uređaj, neto kapaciteta permeata 700 L/h pri optimalnom fluksu od ~30 L/(h m^2). Ispitivane su mogućnosti uklanjanja visokih koncentracija arsena, prirodnih organskih materija preko lako oksidujućih (*COD*) i ukupnih organskih materija (*TOC*), natrijuma i amonijaka (AM) iz influenta pri različitim fluksevima i transmembranskim pritiscima. Nanofiltracijom koncentrata na pilot uređaju je značajno smanjen sadržaj navedenih sastojaka što preko srednjih vrednosti efikasnosti uklanjanja ($\bar{\eta}$) iznosi: $\bar{\eta}_{\mathrm{As}}$ =96,74%, $\bar{\eta}_{COD}$ = 98,23%, $\bar{\eta}_{TOC}$ = 98,73%, $\bar{\eta}_{\mathrm{Na}}$ = 93,45%, $\bar{\eta}_{\mathrm{AM}}$ =80,40%. Na osnovu dobijenih rezultata u radu je predstavljen konceptualni dizajn postrojenja za kondicionairanje vode za grad Kikinda. Srednje vrednosti koncentracija As, TOC, i COD u proizvedenom permeatu su iznosile 2,6 µg/L, 0,33 mg/L i 1.7 mg $KMnO_4$/L, redom, dok su srednje vrednosti za Na i amonijak iznosile 56,66 i 1,14 mg/L, redom. S obzirom na to da su svi mereni parametri u permeatu ispod maksimalno dozvoljene koncentracije može se zaključiti da se primenjene membrane mogu uspešno koristiti za dobijanje pijaće vode iz voda sa visokim sadržajem organskih materija i arsena. Izuzetak je amonijak čija koncentracija u permeatu minimalno premašuje maksimalno dozvoljenu koncentraciju. U radu su takođe ispitivani koncentracioni faktori za svaki mereni parametar. Koncentracioni faktori su određivani teorijski i empirijski, a prikazan je i njihov odnos pomoću kojeg je bilo moguće odrediti pojavu nastajanja naslaga na membranama. Na osnovu prinosa koncentrata i iskorišćenja membrana utvrđen je procenat smanjenja količine otpadne vode. Pri optimalnim vrednostima fluksa i transmembranskog pritiska od ~30 L/(h m^2) i 6,5–7,5 bar, redom, dobijene su vrednosti prinosa koncentrata i iskorišćenja membrana od 8,89% i 90,37%, redom, što označava povećanje iskorišćenja membranskih procesa na oba uređaja zajedno i smanjenja količine koncentrata za ~9%.

Ključne reči: Nanofiltracija • Ušteda vode • Prinos koncentrata • Arsen • Huminske materije • Koncentracioni faktor