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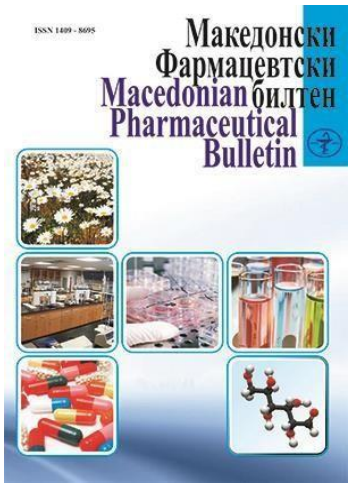

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Comparative analysis of physicochemical and microbiological quality of drinking water before and after ultrafiltration in the city of Štip

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Abstract

Ensuring access to safe drinking water is a global priority. The aim of our study was to evaluate raw water quality collected in the Hydrosystem Zletovica and to compare it with its quality in the water distribution system in the city of Štip, obtained after ultrafiltration (UF) treatment. During 2024, physicochemical parameters color, conductivity, pH, turbidity, potassium permanganate index, and levels of Cl^-, NO_3^-, NO_2^-, NH_4^+ and iron and manganese ions as well as microbiological parameters were analysed in the investigated samples of raw water. The same parameters together with aluminium levels were investigated in the samples from the water distribution system obtained after UF. Results indicate significant improvements in water colour, turbidity and permanganate index in the water distribution system after the ultrafiltration treatment. Using UF, the required quality of drinking water in accordance with the national standards was enhanced.

Key words: drinking water, water treatment, ultrafiltration, water quality

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Introduction

Access to clean and safe water for human consumption was declared a human right by the United Nations General Assembly in July 2016 (UN, 2016). The supply of high-quality drinking water is crucial for human health, the well-being and the overall quality of life as well as the development of modern society (Ren et al., 2023). Drinking water sources primarily come from freshwater inflow such as surface river water, lakes or other water reservoirs (Chew et al., 2015; Davies and Mazumder, 2003). Surface water must be cleaned before usage to remove chemical, biological or physical contaminants in order to be safe for drinking and to meet high quality standards for drinking water. Therefore, these water resources are subjected to various treatment technologies to ensure that water is safe for human consumption by removing impurities from groundwater or seawater (Aziz et al., 2024).

Water treatment technologies depend on the type of raw intake water that comes from various water sources, such as surface water and groundwater (Razali et al., 2023). Ultrafiltration (UF) is a water treatment process for separating, concentrating, and purifying solutions between micro- and nanofiltration via its membrane separation technology and it can be used for applications as diverse as drinking water treatment or biopharmaceutical processing (Drewes et al., 2003). It is one of the most widely used alternative treatment process in potable water production. As one of the most important advantages, UF can replace the entire conventional potable water treatment process of sedimentation and filtration due to its ability to remove different types of particles, colloids, bacteria or viruses (Chew et al., 2015; Mierzwa et al., 2012; Tian et al., 2013).

The multipurpose Hydrosystem "Zletovica" is designed for the utilization of waters from the Zletovska River and its tributaries in the upper course. The process of collecting the surface water is for the water supply of the municipalities of Probištip (Intake 1) and Štip, Lozovo, Karbinci and Sv. Nikole (Intake 2). The water is taken from Zletovska River and it doesn’t pass through any populated place guaranteeing the provision of water from an area where no industrial activity takes place. In this area other possible anthropogenic influence is reduced to a minimum. Upstream the Kneževo Reservoir are the villages of Kneževo, Kojkovo and Emirica, which represent potential polluters although the local population is small and engages in livestock breeding and agriculture (potato cultivation, possible contamination with pesticides and herbicides

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as well as with nitrogen forms). Their influence through the largest tributaries of the Zletovska River - Kučeška and Venečka River, affects the water quality at Intake 1. Around the catchment area of Kučeška and Venečka River, intensive tree cutting and forest road construction is carried out, which contributes to the creation of large erosive areas and represents a potential danger for water pollution especially after heavy rainfall. The collected surface water has high content of phyto- and zoo-plankton and large variations in turbidity, so it is subjected to ultrafiltration treatment to obtain optimal quality drinking water (Public Enterprise Hydrosystem Zletovica website).

The final drinking water quality is the result not only of the treatments used in its purification, but the drinking water distribution system which also plays a crucial role because from the cleaning phase in the ultrafiltration to the point of consumption by the end user, many factors could influence the contamination of the drinking water flowing through the distribution system (Alvares-Arroyo et al., 2015).

Monitoring physicochemical and microbiological parameters of surface water before and after ultrafiltration, as well as in the drinking water distribution system (DWDS) is performed in order to evaluate treatment effectiveness, ensure the production of safe water, protect the expensive UF membranes and downstream equipment, and to comply with environmental and public health regulations (Syeed et al., 2023). The existing studies involve the quality of treated water directly collected after UF rather than that flowing in drinking water distribution systems (DWDS).

The aim of this study was to compare both physicochemical and microbiological quality of water from the Hydrosystem Zletovica with the quality obtained after raw water ultrafiltration treatment and distributed in the DWDS.

Materials and methods

Characteristics of the ultrafiltration process

The facility where the UF is performed is the Filter station of the Public Enterprise Isar in Štip, intended to purify raw surface water from Zletovska River. Before UF, the water is subjected to pretreatment first by adding 1% $Ca(OH)_2$ solution (pH correction), followed by coagulation and sedimentation and then pre-filtration. This procedure is a widely used and effective pretreatment

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method, primarily to adjust pH and hardness as well as to remove impurities like heavy metals and organic matter.

The dosing of chemicals in the pretreatment is based on raw water inlet flow, its pH value and the inlet turbidity. The application of reagents is carried out in distribution chambers after the inlet chamber. The mixing process of reagents for flocculation formation is performed by special mixers.

Coagulation, flocculation and pre-filtration

The coagulant solutions (5% $Al_2(SO_4)_3$ solution or 17% poly-aluminum chloride solution (PAlCl)), prepared in an automatic working unit, are applied based on inlet water turbidity. Flocculation takes place in flocculation basins. Afterwards, the water goes to sedimentation tanks in the form of laminar settlers where sedimentation is performed. When coagulation-flocculation is over the water is led through buffer basins to the prefiltration process. As part of the UF plant, two automatic self-cleaning filters are installed on the inlet pipeline in order to remove coarse particles larger than 150 μm, thereby preventing larger particles from clogging the UF membrane.

Ultrafiltration

The ultrafiltration plant has a nominal capacity of 300 L/s raw water. The technology used is membrane pressure UF with vertical modules, using hydrophilic membrane composed of a blend of polyvinylpyrrolidone and polyethersulfone, with a nominal pore size of 20-30 nm with a high performance and a very good anti-fouling behavior. In this way, all suspended solids, microorganisms and their residues, as well as dissolved organic matter that is absorbed on suspended particles are efficiently removed. During the UF process the following operations take place: filtration, hydraulic washing, chemical washing, and chlorination. During filtration, particles larger than the pores of the membrane are separated. The water flow is established due to the pressure difference on both sides of the membranes and passes through a multitude of small tubes whose wall is the membrane, in the direction from the inside of the tube outward. Impurities remain inside the tube. Colloidal particles, as well as bacteria, parasites, and viruses are also removed. The purified water (filtrate) is collected in a special reservoir. The efficiency of UF is around 90%. Chemical washing is a step that is performed after a certain number of hydraulic washes. The water after washing is led to a neutralization system. After neutralization, the water

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is discharged and led to chlorination with an automatic gaseous chlorine system in order to ensure the required chlorine residual for protection against subsequent water contamination during transport to the end consumer. The determination of residual chlorine is performed on the water at the outlet from the reservoir into the water supply network.

Water sampling

The sampling was carried out according to a previously prepared rigorous protocols to assess water quality (Official Gazette N°18/99, 1999). Sampling was carried out on raw surface water (RSW) and on the water samples on precisely specified locations in the drinking water distribution system (DWDS). These "locations" were selected to monitor the water quality before ultrafiltration, and the quality in the DWDS. This paper presents the results from measurement point at Hydrosystem Zletovica and the selected locations from the DWDS, during 2024. The RSW sampling was performed on 3 measurement points, once a month over the course of 12 months. Also, 6 samples from DWDS were collected weekly over a period of one year.

The samples for physicochemical analysis of water were collected in chemically clean bottles made from inert plastic with volume of 1000 mL. Before sampling, the bottles were rinsed at least three times with the water to be tested. The bottles were filled with the water sample and a small empty space of 10-25 mL was left before closing the bottle. Samples were properly labeled and delivered to the laboratory the same day. Preferably, all analyses were performed on the same day. If the analysis could not be performed, samples were kept in the refrigerator (4 °C) and analyzed within the next 48 hours. Turbid samples were filtered prior analysis, whereas non-turbid samples were analyzed without prior preparation.

For the microbiological analysis, water samples were collected in sterile bottles without added reagents. For the RSW analysis, the samples were collected in a glass bottle with aluminum cap (500 mL) and sterilized at 161 °C, for a period of 2 hours. The sterile bottle was submerged approximately 50 cm below the water level with the opening facing the water flow. For the water sample to be representative, it was collected from the central flow of the water. For the analysis of the chlorinated water samples, 0.3 mL 5% $Na_2S_2O_3$ was added before sterilization to neutralize residual chlorine and ensure a real sample and accurate results (to avoid a false negative result). If there was no possibility for immediate analysis, water samples for microbiological analysis were be kept for a period of 24 hours at a temperature of 5±3 °C (at ambient temperature, testing must begin within 6 hours after sample collection). RSW samples were tested for the presence of: the

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total coliform bacteria, *E. coli*, intestinal enterococci, aerobic mesophilic bacteria at 37 °C as well as for the presence of *Pseudomonas aeruginosa.* The samples from the DWDS were tested for the presence of the total coliform bacteria, *E. coli*, intestinal enterococci, aerobic mesophilic bacteria at 37 °C, *Pseudomonas aeruginosa*, aerobic mesophilic bacteria at 22 °C, fecal coliform bacteria as well as sulfite-reducing clostridia. The investigated microbiological quality parameters for the RSW and for the DWDS with the respective methods used are presented in Table 1. The results from the microbiological analysis were expressed as number of bacteria per 100 mL of water sample.

Table 1

Physicochemical parameters

The physicochemical analysis for the RWS comprised of determination of the following parameters: color, conductivity, pH, turbidity, permanganate index and chloride (Cl^-), nitrate (NO_3^-), nitrite (NO_2^-), ammonium (NH_4^+), iron (Fe), and manganese (Mn) levels.

The color (expressed on a Pt/Co scale) and the levels of nitrate (NO_3^-), nitrite (NO_2^-), ammonium (NH_4^+), iron (Fe), and manganese (Mn) were determined by using an ultraviolet-visible spectrophotometer (PROVE 300, Merck, Germany). Chloride (Cl^-) levels were quantified using Mohr's method, involving silver nitrate and potassium chromate as an indicator (MKC EN ISO 9297:2007). The permanganate index was determined according to the method MKC EN ISO 8467:2007. In brief, the principle of the method is heating a sample with a known amount of $KMnO_4$ and sulfuric acid for a fixed period time (10 min) in a boiling water-bath, reducing part of the permanganate by oxidizable material in the sample and determining the consumed $KMnO_4$ by addition of an excess of oxalate solution, followed by titration with $KMnO_4$.

Turbidity of the samples was measured using a Lovibond TB 300 IR turbidimeter (Tintometer, Ltd., Germany). The results were expressed as nephelometric turbidity units (NTU) according to the MKC EN ISO 7027:2017. The pH-value determination as well as the conductivity measurements were performed according to the MKC EN ISO 10523:2013 and MKC EN 27888:2007, respectively (MKC EN ISO 10523:2013; MKC EN 27888:2007). The pH-value determination was performed on a Mettler Tolledo pH meter (Mettler Tolledo, Germany). The ISO method used for this determination is a method for determining the pH value in rain, drinking and

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mineral waters, bathing waters, surface and ground waters, as well as municipal and industrial waste waters, and liquid sludge, within the range pH 2 to pH 12 (ionic strength below I = 0.3 mol/kg and conductivity < 2 000 mS/m at 25 °C) and in the temperature range 0 °C to 50 °C. The method specified for conductivity determination is suitable for all types of water including surface waters, process waters in water supply and treatment plants, and waste waters. The measurement was performed on a Mettler Toledo conductometer (Mettler Tolledo, Germany).

To ensure the reliability of the analytical results, all measurements were performed in triplicate. Calibration of all used instruments was regularly conducted using certified reference materials.

Statistical analysis

Statistical analysis was conducted using the Microsoft Excel. All the data were processed to obtain descriptive statistics, minimums and maximums in order to get an overview of the data collected and their variability.

Results and discussion

Efficient use of water resources in water treatment facilities requires a clear knowledge of surface water quality and its variability since it influences the proper treatment choice (Mensah - Akutteh et al., 2022; WHO, 2019). In this study, the physicochemical and microbiological parameters of the RSW samples and the DWDS samples after UF during 2024 were analyzed. The summary of results for the physicochemical parameters from the RSW are presented in Table 2.

Table 2.

As it can be seen from Table 2, most of the investigated parameters were in accordance with the national regulatory requirements (Official Gazette for Drinking Water Quality, 2018). This is a relatively clean water. All the investigated parameters were below the maximum allowable limit, except for the turbidity and the consumption of $KMnO_4$. Turbidity occurs due to the presence of soil, organic, and inorganic matter, plankton, and other microscopic organisms. The turbidity values ranged from 1.09 NTU to 8.69 NTU exhibiting fluctuations being well above

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the permitted level of 1.50 NTU. The highest value was observed in October while the lowest turbidity values were registered in January. It is expected that RSW turbidity values will be higher since RSW contains suspended matter such as clay, silt, finely dispersed organic and inorganic matter, soluble colored organic compounds, plankton, and other microscopic organisms.

The permanganate index measures the oxygen equivalent of substances oxidized by potassium permanganate under specific conditions. It serves as an aggregate indicator of pollution by organic and reducing inorganic substances (Du et al., 2024; Zhang, 2008). Under specified conditions, potassium permanganate, a strong oxidizing agent, oxidizes the reducing substances in the water sample. After the reaction, excess oxidizing agent is reduced using sodium oxalate, and the consumption of potassium permanganate is calculated by back-titration, expressed in milligrams per liter of oxygen (Jianxun and Haiqiong, 2025). The permanganate index ranged from 4.44 to 10. 39 mg/L oxygen with the highest values observed in December and the lowest in March.

Nitrate (NO_3^-) and nitrite (NO_2^-) are naturally occurring ions that are ubiquitous in the environment. Both are products of the oxidation of nitrogen, as part of the cycle required by all living systems for the production of complex organic molecules, such as enzymes and other proteins (Environment Canada, 2003; IARC, 2010; WHO, 2016). They serve as key pollutants and indicators of water quality and their accurate detection is essential for monitoring and maintaining water safety (Ingles et al., 2021; Mai et al., 2025). About 50% of nitrogenous fertilizers used in agriculture end up leaching or draining into surface and groundwater, leading to elevated nitrate and nitrite levels in natural water bodies. According to the World Health Organization, nitrate levels in drinking water should be below 50 mg/L and nitrite levels under 3 mg/L (expressed as nitrate and nitrite ions, respectively) to prevent health issues such as methemoglobinemia, thyroid problems, and risks to bottle-fed infants (WHO, 2017). Therefore, real-time monitoring of nitrate and nitrite levels in surface water and rainwater is important to capture better data and gain a better understanding of nutrient dynamics and environmental impacts. As it can be seen from the results shown in Table 2, NO_3^- levels in the RSW were within the required values of the national regulative and consistent with the WHO standards for drinking water. The NO_2^- levels in the investigated samples were in the range of undetectable levels to 0.16 mg/L as a maximal level measured in August.

Ammonium (NH_4^+) is an inorganic compound present in water at pH < 9. It has no toxic effects on human health, but its presence in water can indicate a water pollution. It is reported that

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ammonium can be found only in polluted waters (Radu and Racoviteau, 2021). The highest observed value for NH_4^+ ion concentration was 0.19 mg/L, well below the maximal allowable limit of 0.50 mg/L.

Chloride ions (Cl^-) are widely distributed in nature. Natural water contains Cl^-, mainly in the form of calcium, magnesium, and sodium salts. Natural water samples from different sources vary in Cl^- content. The Cl^- content in seawater can be as high as 19,000 mg/L, while the Cl^- content in general surface water and ground water is less than 50 mg/L. The chloride ion is one of the major pollutants in drinking water, which causes a wide spectrum of biological and environmental impacts in aquatic and terrestrial ecosystems (Moradi et al., 2023). In our study, the Cl^- levels were low and steady throughout the entire year without major fluctuations.

Iron and manganese are found naturally in several soil and rock minerals; the main ones are hematite (Fe_2O_3) and pyrolusite (MnO_2) and they reach surface waters when the water is dissolving these minerals in the surface strata. Surface water does not usually contain high concentrations of iron or manganese because the oxygen-rich water enables both minerals to settle out as sediments (Inglezakis et al., 2010). The average iron level in the studied samples of RSW were below the maximal allowable limit of 0.20 mg/L, except for one measurement being above allowable limit (0.55 mg/L). The manganese levels were below the detection limit all year long.

The results from the comparative analysis between samples from RSW and samples from the DWDS obtained after UF are presented in Fig. 1 and Fig. 2. As shown in Fig. 2, no significant changes were observed for conductivity, pH and the levels of Cl^-, NO_3^- , NO_2^{-*}, NH_4^+ and iron and manganese ions. This is expected as the pores of UF membrane are big enough to allow the passage of free ions. After the UF, there is a noticeable difference in the parameters colour (Fig. 1 A), turbidity (Fig. 1 B), and permanganate index (Fig. 1 C). The turbidity levels in the samples taken from the DWDS range from 0.34 NTU to 0.56 NTU and are constant throughout the entire year. This significant drop in turbidity levels is due to the UF process. The consistent trend of lowered turbidity values suggests efficient removal of suspended particles, solids or organic matter (Aoueryagel et al., 2024, Mayer et al., 2025). The results for the parameter colour in the DWDS indicate marketed improvement after treatment which suggests effective removal of colour causing substances. These results align with the findings from the study performed by Sandoval-Olvera et al. (2019) which reported similar results using UF systems combined with coagulation pre-treatment (Ghonimy et al., 2025). There is a visible difference for the parameter permanganate

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index as well. However, in this case, the reduction is a result of coagulation-flocculation pre-treatment than the ultrafiltration itself (Laszakovits and MacKay, 2023). Aluminium levels were determined in the samples from the DWDS, since aluminium compounds are used for the RSW pre-treatment. The obtained values for aluminium levels in the investigated samples ranged from 0.040 - 0.056 mg/L, levels consistent with the national regulatory requirements.

Fig 1

Fig 2

The evaluation of the microbiological parameters is one of the most important segments in water quality assessment. In our study, intestinal enterococci, *E. coli* and total coliform bacteria were detected in RSW samples and *P. aeruginosa* was detected in RSW samples in August only. Total aerobic mesophilic bacteria at 37 °C were absent from the investigated samples. After the UF treatment, the investigated samples from the DWDS showed no detectable levels of the microbial indicators, showing effective microbial removal by UF and improved microbiological quality. In addition, ultrafiltration membranes with a typical pore sizes of 20-30 nm effectively retain bacteria, large cells as well as viruses. As a result, UF can physically disinfect water thereby contributing to hygienization (Meyer et al., 2025).

Conclusion

In this study, a comparative quality assessment of samples from RSW and DWDS was performed. The efficiency of the UF process in improving water quality was evaluated through analysis of various physicochemical and microbiological parameters. The obtained results showed significant improvement in the parameters turbidity, color, and permanganate index after UF. All bacteria were also removed. The UF of surface water proved very useful for drinking water production. Few sensory characteristics of UF water were significantly improved, along with the fulfillment of the regulatory requirements. Therefore, advanced treatment methods such as UF are needed in order to satisfy the growing demands for clean water. However, limited resources especially in low- and mid-income countries remain the main challenge for routine use of UF.

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Резиме

Компаративна анализа на физичко-хемискиот и микробиолошкиот квалитет на водата за пиење пред и по ултрафилтрација во градот Штип

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Клучни зборови: вода за пиење, третман на вода, ултрафилтрација, квалитет на вода

Обезбедување пристап до безбедна вода за пиење претставува глобален приоритет. Целта на нашето истражување беше процена на квалитетот на сировата вода за пиење собрана во хидросистемот Злетовица и негова споредба со квалитетот на водата за пиење од водоводната мрежа на градот Штип добиена по ултрафилтрација (УФ). Во 2024 година, физичко-хемиските параметри боја, спроводливост, pH, матнотија, потрошувачка на калиум перманганат и концентрациите на Cl, NO_3^-, NO_2^-, NH_4^+, концентрациите на железни и јони на манган, како и микробиолошките параметри беа испитани во примероци на сирова вода. Истите параметри, заедно со параметерот концентрација на алуминиумови јони беа испитани во примероци од водоводната мрежа, добиени по УФ. Резултатите покажаа значително подобрување кај параметрите боја, матнотија и потрошувачка на калиум перманганат во примероците земени по УФ од водоводната мрежа. Со примена на оваа

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постапка, квалитетот на водата за пиење беше подобрен, во согласност со националните стандарди.

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Table 1. Microbiological quality parameters in RSW and DWDS with their respective methods

Raw surface water (RSW)	DWDS	Method
Intestinal enterococci	Intestinal enterococci	MKC EN ISO 7899-2:2000
Pseudomonas aeruginosa	*Pseudomonas aeruginosa*	MKC EN ISO 16266:2009
Aerobic mesophilic bacteria at 37 °C	Aerobic mesophilic bacteria at 37 °C	MKC EN ISO 6222:2009
Total coliform bacteria	Aerobic mesophilic bacteria at 22 °C	MKC EN ISO 6222:2009
Esherichia coli	*Esherichia coli*	MKC EN ISO 9308-1:2015
	Fecal coliform bacteria	MKC EN ISO 9308-1:2015
	Sulfite-reducing clostridia	MKC EN ISO 14189-1:1993
	Total coliform bacteria	MKC EN ISO 9308-1:2015

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Table 2. Summary of the results for the RSW physicochemical parameters during 2024

Parameter	Units	MIN	MAX	Surface water quality standard*	
				Minimum allowable limit	Maximum allowable limit
Color	Pt/Co	1.60	2.90	/	20
Conductivity	µS/cm	75.40	126.50	/	2500
pH	/	7.46	7.74	6.50	9.50
Turbidity	NTU	1.09	8.69	/	1.50
$KMnO_4$	mg/L	4.44	10.39	/	8
Cl^-	mg/L	4.00	8.00	/	250
NO_3^-	mg/L	0.67	2.13	/	50
NO_2^-	mg/L	BDL*	0.16	/	0.10
NH_4^+	mg/L	BDL	0.19	/	0.50
Fe	mg/L	BDL	0.55	/	0.20
Mn	mg/L	BDL	BDL	/	0.05

*BDL=below detection limit, interpreted using the regulation criteria for drinking water

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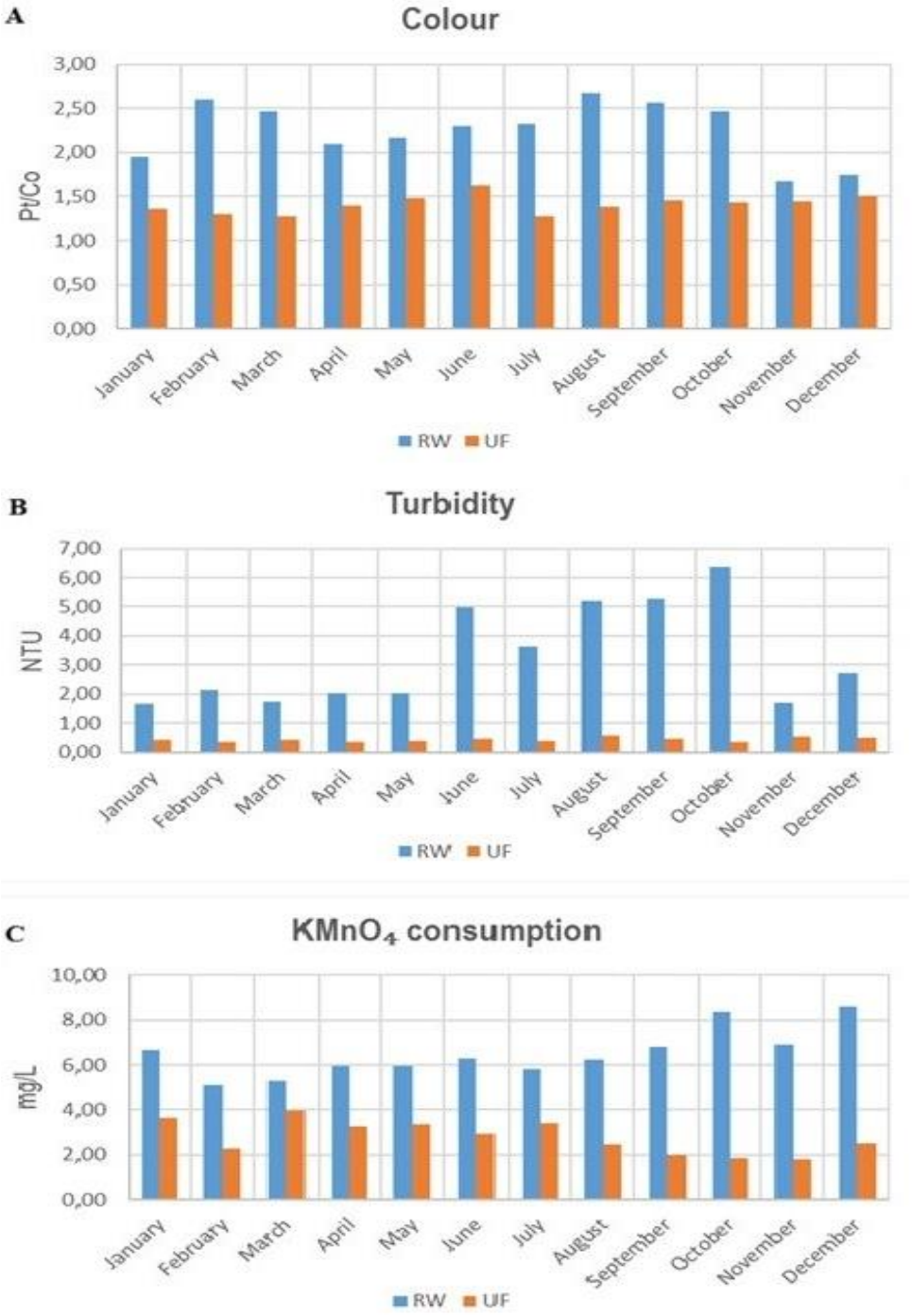

Fig. 1. Comparative analysis of physicochemical parameters between RSW (blue) and DWDS (orange) samples: A) Colour; B) Turbidity; C) Permanganate index.

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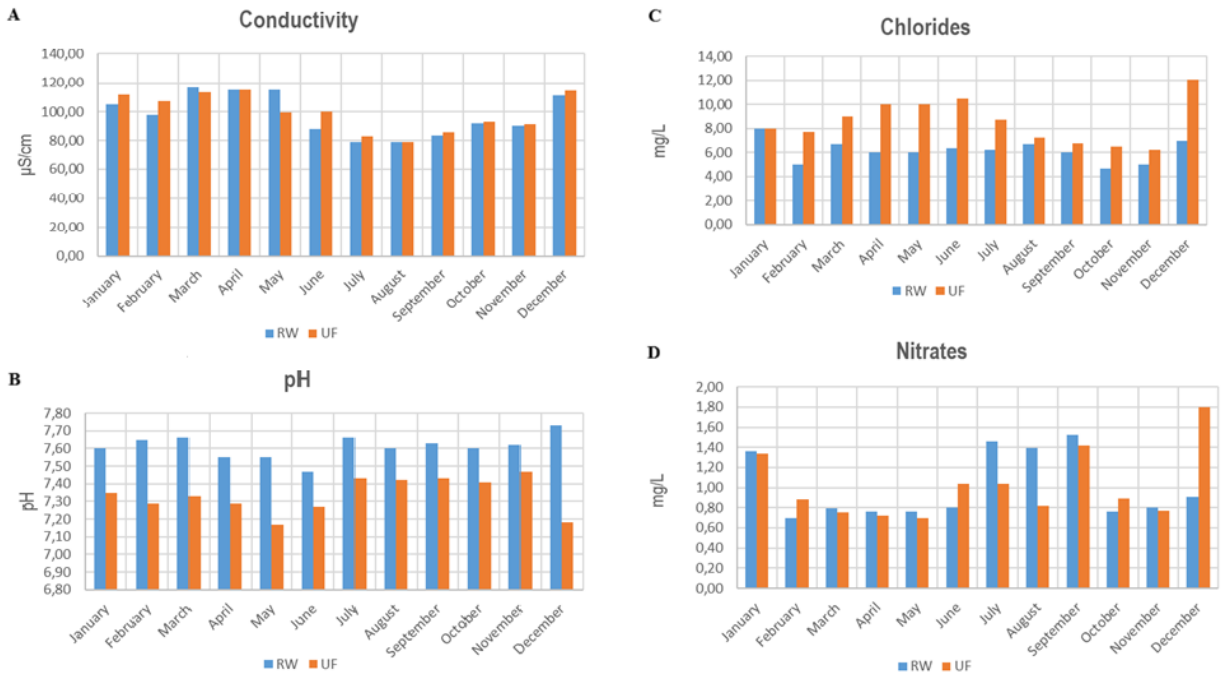

Fig. 2. Comparative analysis of physicochemical parameters between RSW (blue) and DWDS (orange) samples: A) Conductivity; B) pH; C) Chlorides D) Nitrates.

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