

Water Softening Systems of Low and Medium Capacity

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ABSTRACT

The current state of the hydrosphere is largely determined by human anthropogenic activities. Discharge of polluted waste waters into surface water bodies significantly worsens their quality and limits the possibility of safe consumption. One of the biggest sources of increasing surface waters mineralization and hardness is the discharge of spent regeneration solutions of natural waters ion-exchange softening processes, which creates a closed circulation cycle of sodium, calcium and magnesium chlorides. The necessity to reduce the intensity of this cycle and decrease environmental pollution is very actual today. The research of traditional soda-sodium softening technology has shown its low suitability for low and medium capacity systems due to necessity of water heating and subsequently pH adjusting. More acceptable solution was found in using phosphate anions as a precipitant, which ensures high efficiency in removing calcium and magnesium ions at a wide range of temperatures and pH levels. It was found that the solid phase formed during such treatment was difficult to separate from the liquid phase. Using of anionic flocculants was proposed as one of the solutions of this problem, which ensure high efficiency in separating the formed solid particles from water. Proposed technological scheme was designed for implementation as a pre-treatment stage in reverse osmosis systems of low and medium capacity.

Keywords: water softening, water hardness, water treatment, calcium ions, magnesium ions, water treatment by phosphates, flocculants, filtration, domestic softening systems.

INTRODUCTION

Direct water consumption from most surface water bodies in Ukraine today is practically impossible due to its bad quality, which isn’t respond to regulatory standards (Radovenchyk et al., 2022, Alipov et al., 2007, Goncharuk et al., 2001). The main parameters that do not comply with current requirements are water mineralization and hardness. The exceeding of these parameters hinders human consumption and possibility to use at industrial enterprises. Eastern and Southern regions of Ukraine are particularly affected by this situation, where we have a significant discharge of highly mineralized mine waters into surface water bodies. In these regions, the water mineralization and hardness are constantly

increase. These problems have also affected groundwater, which in some regions of Ukraine also exhibits elevated levels of mineralization and hardness. As researches show, excessive intake of calcium and magnesium ions into the human body can negatively affect the musculoskeletal system and cardiovascular system, calcium and magnesium compounds can accumulate as stones in the kidneys and gallbladder. The significant depletion of quality natural water resources highlights the issue of water softening, making this problem increasingly relevant and urgent.

The presence of calcium and magnesium ions in water determines a parameter known as hardness. Ukrainian regulatory documents limit the hardness in water for human consumption to 7 mg-eq/dm³ (DSanPiN 2.2.4-171-10, 2010). For

the food industry, water with hardness of 0.1–0.2 mg-eq/dm³ is required, while in the energy sector, the acceptable hardness level is considered in the range of 0.03–0.05 mg-eq/dm³ (Orlov, 2014, Dolinsky et al., 2005). The oldest technology for regulating water hardness is the soda-lime method. Its essence lies in the two-stage treatment: at the first stage sodium carbonate is used to precipitate calcium ions, and then at the second stage is used lime to precipitate magnesium ions (Zapolskyi, 2005, Hasson and Cornel, 2017). After separating the solid phase by settling or filtration, at the treated water the pH level is adjusted to neutral. After the described stages, the residual hardness of the treated water is 0.5–1.0 mg-eq/dm³. For the effective reaction, the water temperature needs to be maintained at 35–40 °C. Today this technology is widely used in industry, although many aspects of the softening process have not been fully explored yet.

The most acceptable and progressive process today is water post-treatment directly at points of water consumption. Therefore, water softening systems are increasingly being installed and operated in private houses, apartments and offices. The most convenient systems are based on ion exchange technology (Fischer et al., 2023, Homelia et al., 2008; Tykhonova and Matsiievska, 2004). These systems can operate automatically for extended periods and require minimal maintenance. However, one of the drawbacks of their operation is the formation of regeneration solutions with high concentrations of sodium chloride and significantly lower concentrations of calcium and magnesium ions. Additionally, 20–30% of the treated water is used for internal needs, such as washing and ion exchange resins regeneration (Trus et al., 2016, Ayrapetyan, 2010, Nazarenko, 2015). As a result, the operation of such systems returns not only the calcium and magnesium ions to surface water bodies, but also a lot of sodium chloride compounds (Shablilii et al., 2014). It can be anticipated that the existence of such a cycle will soon cause a need to softening desalinating treatment of natural water. Investigating the possibility of breaking this cycle is extremely important today.

In industrial water softening technologies, generated waste is disposed or accumulated in special areas. For low and medium capacity systems, such procedures are too expensive and are not currently provided for. One potential solution to this problem could be in changing ion exchange process with a more simpler

reagent method. According to current regulatory documents (DSanPiN 2.2.4-171-10, 2010), drinking water should contain 25–75 mg/dm³ of calcium ions and 10–50 mg/dm³ of magnesium ions. Therefore, it makes sense to explore the efficiency of the reagent method for use in private houses and offices using different precipitants. In industry, this method is mainly implemented at increased water temperatures, with extended settling process to form a solid phase, and it is generally characterized by relatively low efficiency. Scientific literature has no data on the efficiency of reagent technologies under real hydrosphere temperatures and immediately using after reagent treatment. Therefore, the goal of our experiments was to study the specifics of reagent softening under various conditions, considering the possibility of its application in household and office systems.

MATERIALS AND METHODS

Materials

The objects of the study were solutions with a total hardness level of 5–35 mg-eq/dm³ (with varying concentrations of magnesium and calcium ions), which simulated natural waters from surface and groundwater sources, as well as waters from centralized water supply systems.

Removal of hardness ions from water

During the study the 0.1 dm³ of the model solution was treated with the appropriate volume of the precipitating reagent to convert Ca²⁺ and Mg²⁺ ions into insoluble compounds and separate them from the treated water. If necessary, the temperature and pH of the initial model solution were adjusted. After adding the reagents, the suspension was settled in graduated cylinders, and corresponding graphs of the change of apparent sediment volume over time were recorded. To evaluate the filtration properties of the formed sediment, a standard funnel with a diameter of 60 mm and a 110 mm diameter “blue ribbon” paper filter was used, placed above the 0.1 dm³ volume graduated cylinder. The filtration rate was determined while maintaining the maximum suspension level in the funnel when separating a 0.1 dm³ suspension. The hardness of the initial and treated solutions was determined by titration using the Eriochrome Black T (Chromogen Black T) and Trilon B solution (Li et al., 2013).

RESULTS AND DISCUSSION

Our experiments showed that effective precipitation of calcium ions by sodium carbonate from dilute aqueous solutions requires a significant excess of the precipitant compared to the stoichiometric amount (Radovenchyk et al., 2022). For example, at a component ratio of $K = [\text{CO}_3^{2-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}] = 1$, the method's efficiency at low water temperatures is extremely low. Measurements of water temperature in Kyiv's water supply system showed fluctuations within the range of 13–17 °C, and in the winter period, it can decrease to 8.5 °C. Therefore, water temperature within this ranges was chosen for further experiments. It was found that when water is treated with an equivalent amount of precipitant, the hardness is reduced only 4%, and after an hour of settling, this parameter reaches 17%. At the same time, the formation of a solid phase is not visually observed, although a decrease in hardness over time during filtration by “blue ribbon” filter is noted. Increasing the precipitant dose provides slightly better results. However, the reagent consumption is not commensurate with the achieved effect. For example, a fivefold increase in the sodium carbonate dose only halves the total hardness of the treated water, while a tenfold excess of the stoichiometric ratio provides residual hardness at the level of 0.5–0.6 mg-eq/dm³. Further increasing the precipitant dose has virtually no effect on the residual hardness of the treated water.

At the coefficient values $K = [\text{CO}_3^{2-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}] > 2$, the addition of sodium carbonate begins to be accompanied by cloudiness

of the solution and the formation of a solid phase, which can be visually observed. Initially, there is a barely noticeable cloudiness of the treated water at $K = 3$, followed by the formation of clearly visible solid particles at $K = 5$, and intensive sediment formation after the addition of the precipitant at $K = 8$ –10. The formation of sediment allows the study of the clarification characteristics of the treated water, which is often used for separating the solid phase. It should be noted that at such low temperatures, the clarification process is quite unstable. There is no clear correlation between settling intensity and the component ratio. Considering that the precipitation conditions are identical in all cases, it can be assumed that calcium carbonate compounds of varying structure and composition are being formed.

As the results of further studies have shown (Fig. 1), the water temperature significantly affects the calcium ion precipitation process. Since heating water above 70 °C requires considerable energy costs, the water temperature in our studies was limited at the range of 10–70 °C. It was found that water softening is more effective with higher initial hardness level. As seen in Fig. 1, increasing the initial hardness above 8 mg-eq/dm³ allows for calcium ion content to reach 0.6–1.0 mg-eq/dm³ after treatment with the appropriate amount of sodium carbonate. It should also be noted that the formation of the solid phase occurs somewhat differently (Hordiienko et al., 2022).

Thus, at an initial hardness of 4.4 mg-eq/dm³, it is difficult to detect the formation of sediment at low temperatures, and individual flakes form only at temperatures above 40 °C. At an initial hardness of 7.6 mg-eq/dm³, the solution becomes

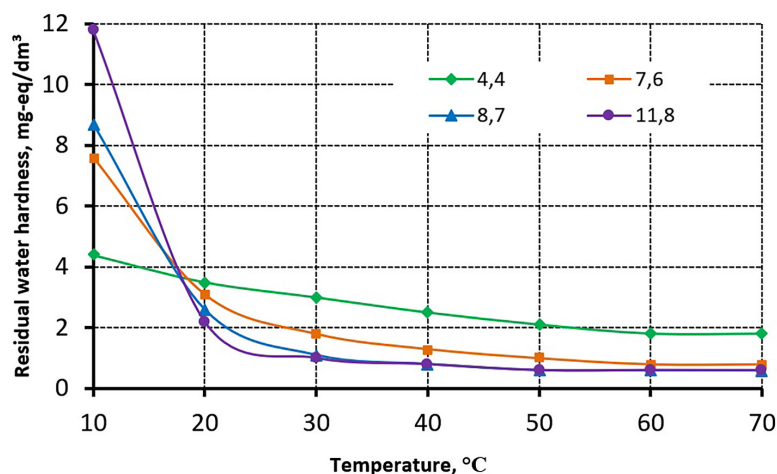


Figure 1. The dependence of residual water hardness on temperature at different initial hardness values (pH = 6.85, coefficient $K = [\text{CO}_3^{2-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}] = 10$)

cloudy when heated up to 40 °C even without a precipitant, and the addition of sodium carbonate is accompanied by the intensive formation of solid particles. With a further increase in the initial water hardness, intense solid phase formation and sedimentation to the bottom of the container are observed with the addition of sodium carbonate, starting at temperature of 20 °C.

As the results of further studies have shown, even at a water temperature of 13 °C, calcium ions precipitate quite intensively under certain conditions. The determining factor is the ratio between the concentrations of carbonate ions and calcium ions, $K = [\text{CO}_3^{2-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}]$. This is particularly noticeable in the range of coefficient K value from 0 to 15.

The research of magnesium ions removal showed that, unlike calcium carbonate, the effective precipitation of magnesium ions is possible in the form of other insoluble compounds (Radovenchyk et al., 2022). According to (Ciganok et al., 2014), the most common water-insoluble compounds are magnesium compounds with phosphorus, and there are many of them with a wide range of solubility. However, the simplest and most widespread is the hydrolysis product of magnesium ions – magnesium hydroxide. It is in the form of hydroxide that magnesium ions are removed in the soda-lime technology, as magnesium carbonate has too high solubility. On the other hand, the pH value for the sedimentation of $\text{Mg}(\text{OH})_2$ precipitation, at an initial magnesium ion concentration of 0.01 mg/dm³, is 10.4, while the pH for complete precipitation is 12.4 (Ciganok et al., 2014). Our research showed that the removal of magnesium as hydroxide is associated with significant disadvantages, because such a significant pH increasing requires subsequent correction after sediment separation, which involves

considerable reagent consumption. Even at an initial hardness of 19.6 mg-eq/dm³, no solid phase formation in the volume of the model solution is visually observed until a pH range is reached at 10.5 level (Fig. 2). For a noticeable softening effect, the pH must be maintained at 11–11.5. On the other hand, the positive aspect is that the clarification of the treated solution is observed, allowing the solid and liquid phases to be separated. However, this method is completely unsuitable for household conditions, as it requires an additional pH correction stage for the treated water and involves significant reagent consumption.

Therefore, further studies were devoted to examining the effectiveness of removing calcium and magnesium ions by treating water with sodium phosphate (Radovenchyk et al., 2022). The methodology remained the same as in the previous experiments. The conducted studies showed that, unlike calcium carbonate, effective precipitation of calcium ions by phosphate is possible even at reagent doses lower than stoichiometric. For instance, at a component ratio of $K = [\text{PO}_4^{3-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}] = 1$, the method's efficiency is quite high at normal temperatures (Fig. 3).

Even at $K = 0.5$, the calcium ion content decreases by more than a half. At stoichiometry ($K = 1$), the residual concentrations of calcium ions in the treated water decreased below 1 mg-eq/dm³. For drinking water such level of calcium ions is even necessary to ensure the physiological completeness of the mineral composition. Another advantage is that the method is high efficiency and is maintained over a wide pH range from 5 to 11. At $K > 1.5$, the calcium ion content in the treated water stabilizes at 0.2–0.1 mg-eq/dm³. Thus, unlike the soda-lime method, which requires significant reagent overconsumption, using phosphates in the softening process allows for the use of stoichiometric reagent

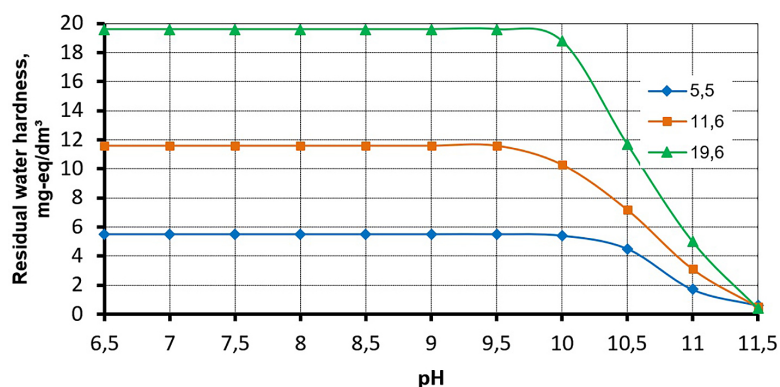


Figure 2. The dependence of residual water hardness on pH at different initial hardness values and solution temperature of 22 °C

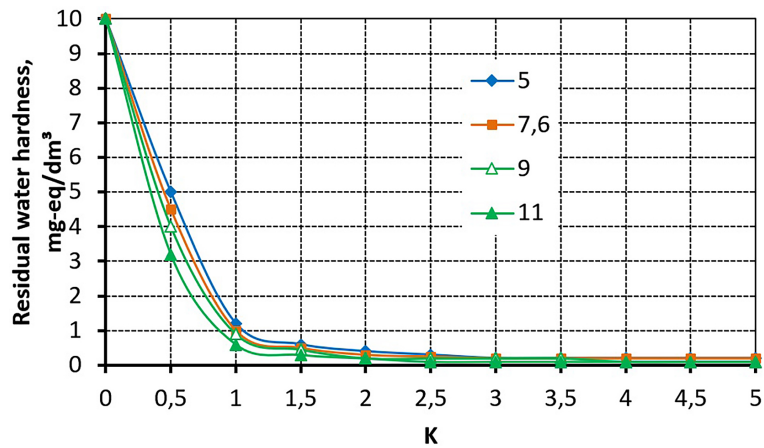


Figure 3. The dependence of residual water hardness on the component ratio $K = [\text{PO}_4^{3-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}]$ at temperature of 20 °C, an initial hardness $H_0 = 10 \text{ mg-eq/dm}^3$ and different initial pH values

ratios. The most important advantage of calcium ion precipitation with phosphates, in line with the goals of our study, is the immediate formation of a solid phase after mixing the components. Upon adding the reagent to the model solutions, immediate clouding is observed, and solid particles form intensively, which after 3–5 seconds begin to aggregate and settle at the bottom of the vessel.

Treated water temperature is an extremely important parameter of the softening process. When sodium carbonate is used as a precipitant, the efficiency of the process at temperatures below 35–40 °C is extremely low, requiring obligated water heating. On the other hand, water in centralized water supply systems and from underground horizons can, at certain times, have a temperature of 10 °C or even lower. Our research of using sodium phosphate as a precipitant, showed that sufficient efficiency was achieved over a wide temperature range (Fig. 4). A noticeable decrease of efficiency is observed only at temperature of 5 °C. However, even in this case, this parameter stands

in ranges from 0.1 to 1.5 mg-eq/dm³, which is quite acceptable for household and office conditions. It is clear that the stability of the softening process across such a wide temperature range allows the use of phosphates in water softening processes as a perspective and efficient method.

The reduction of magnesium hardness using phosphates was proved to be less effective than in the case of calcium ion removal (Fig. 5) (Radovenchuk et al. 2022). Even at pH of 10.07 and $K = 2$, the minimal residual water hardness did not decrease below 0.75 mg-eq/dm³ (for calcium it was 0.1–0.2 mg-eq/dm³). A stable efficiency decreasing is observed with a decrease of pH, although not very significant. Thus, when transitioning from an alkaline to an acidic environment, the residual concentration of magnesium ions doubles regardless of the value of the K coefficient. Given the current strict regulations on phosphate content in wastewater, it is important to focus on stoichiometric ratios of reagents to obtain more complete reaction between components. As

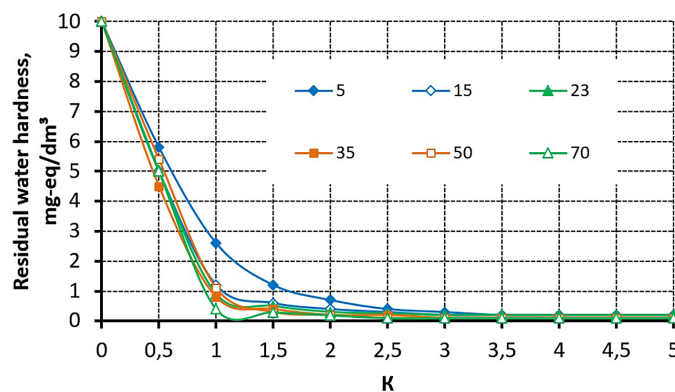


Figure 4. The dependence of residual water hardness on temperature and the component ratio $K = [\text{PO}_4^{3-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}]$ (initial hardness 10 mg-eq/dm³, pH = 7.45)

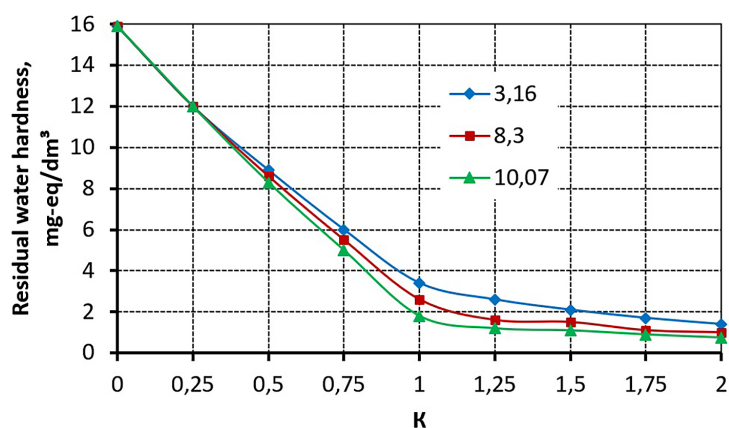


Figure 5. The dependence of residual water hardness on the coefficient $K = [\text{PO}_4^{3-}, \text{mg-eq}] / [\text{Ca}^{2+}, \text{mg-eq}]$ at different pH values and solution temperature of 22 °C

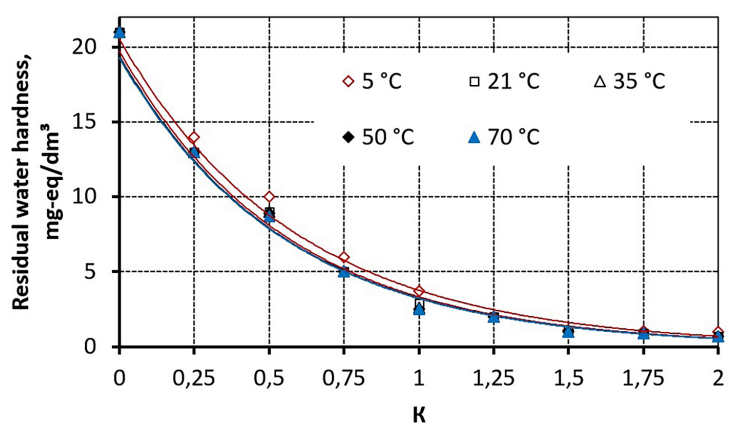


Figure 6. The dependence of residual water hardness on the coefficient $K = [\text{PO}_4^{3-}, \text{mg-eq}] / [\text{Mg}^{2+}, \text{mg-eq}]$ at different solution temperatures (initial hardness = 21 mg-eq/dm³, pH = 6.58)

seen in Figure 5, in a neutral environment with $K = 1$, the residual hardness of the treated water can reach 3 mg-eq/dm³ or even more. Under other conditions (such as temperature or high initial magnesium hardness), the residual hardness may be even higher, significantly reducing the overall efficiency of the softening process.

A significant advantage of sodium phosphate as a magnesium ion precipitator is the fact that water temperature has virtually no effect on process efficiency at a wide temperature range. As can be seen from Figure 6, the softening efficiency remains stable in the temperature range of 5–70 °C. Moreover, the solid phase is formed immediately after the solutions are drained.

The overall effectiveness of reagent-based softening methods can only be ensured by the complete separation of solid and liquid phases. This can be achieved in two ways – filtration or settling. Filtration is more suitable method for low-capacity systems, such as household or office systems, where it is not always possible to install large-volume

tanks. Settling is more appropriate for industrial high-capacity systems with necessary volume settling tanks, which can help to indicate the degree of particle formation in the solid phase.

Given the dispersion of the solid phase formed during phosphate treatment, the removal of solid particles from aqueous solutions through filtration occurs rather slowly, which significantly limits the productivity of the softening technology. Therefore, we investigated the possibility of using flocculants as additional reagents. The following reagents were used: polyacrylamide (PAA) as a nonionic flocculant, Magnofloc-336 by Ciba as an anionic-type flocculant with a molecular weight up to 20 million, and Zetag-7692 by Ciba as a cationic-type flocculant with a molecular weight up to 20 million.

As a result of our research, it was found that the best results were achieved by using the anionic-type flocculant Magnofloc-336 by Ciba in doses of 1–10 mg/dm³ at an initial water hardness 27 mg-eq/dm³ and equal concentrations of calcium

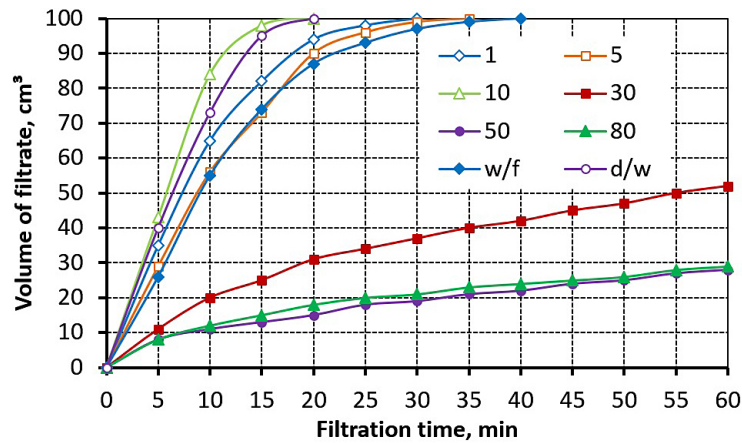


Figure 7. The dependence of the suspension filtration speed over time at $K = [\text{PO}_4^{3-}, \text{mg-eq}] / [\text{Ca}^{2+} + \text{Mg}^{2+}, \text{mg-eq}] = 1$, with different concentrations of Magnofloc-336 (mg/dm^3) (solution temperature = 21°C , initial hardness = $27.0 \text{ mg-eq}/\text{dm}^3$, $[\text{Ca}^{2+}] = 13.5 \text{ mg-eq}/\text{dm}^3$, $[\text{Mg}^{2+}] = 13.5 \text{ mg-eq}/\text{dm}^3$, $\text{pH} = 7.4$) (w/f – without flocculant, d/w – distilled water)

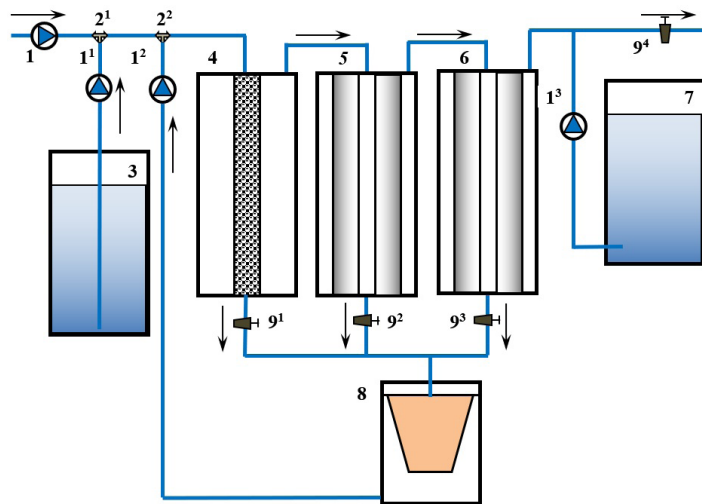


Figure 8. Technological scheme of water softening using phosphates: 1, 1¹, 1², 1³ – pump; 2¹, 2² – injector; 3 – precipitating solution tank; 4 – reagent mixing block; 5 – mechanical filter (20 μm); 6 – mechanical filter (5 μm); 7 – softened water tank; 8 – bag filter; 9¹, 9², 9³, 9⁴ – solenoid valve

and magnesium ions (Fig. 7). Under optimal conditions, the filtration speed of suspension is close to the filtration speed of distilled water.

Other flocculants did not provide a similar effect at any dose and initial water hardness (Radovenchyk et al., 2022, Hordiienko and Radovenchyk, 2023).

Based on the obtained results, a composition of the precipitating mixture for water softening for small and medium-capacity systems was developed. In addition to using flocculants as auxiliary reagents, we proposed to change sodium phosphate (Na_3PO_4), which increases the hydrogen index (pH) of the treated water, to an equivalent mixture of $[(0.75 \div 0.85) \text{Na}_3\text{PO}_4 + (0.25 \div 0.15) \text{NaH}_2\text{PO}_4]$. When using this mixture, the

softened water has low residual hardness, and its pH does not exceed 8.5 (Patent No. 154768, Ukraine, 2023). It is evident that the flocculant dosage and the ratio between the components of the mixture depend on the initial water hardness, calcium and magnesium ions concentration and other characteristics of the treated water. Therefore, the optimal doses and component ratios are selected for each types of water composition.

The conducted research formed the basis for developing a technological scheme of water softening using phosphates. The technology consists of such processes (Fig. 8).

The water is pumped by pump 1 to the softening system to maintain consistent flow characteristics. Using injector 2¹, the precipitating solution

from tank 3 is dosed into the water flow by pump 1¹. The resulting mixture passes through the reagent mixing block 4, which has a perforated tube for additional reagent mixing and for the maturation of particles in the formed solid phase. The resulting suspension passes through mechanical filter 5 with a 20 µm pore size and mechanical filter 6 with a 5 µm pore size. The treated water is supplied to consumers or can be stored in the tank 7.

During the water softening process, the precipitating solution is delivered into the main pipeline by pump 1¹. The formed solid phase is retained in the mixing block 4 and mechanical filters 5 and 6. To ensure the effective operation of the softening system periodic cleaning of blocks 4–6 from the accumulated solid phase is provided. For this procedure, the water supply pump 1 is periodically turned off, solenoid valves 9¹–9³ are opened, and valve 9⁴ is closed. Then, blocks 4–6 are flushed with a reverse water flow using pump 1³, and the obtained suspension flows into the tank with the bag filter 8. The liquid phase accumulates in the lower part of block 8, and when a certain level is reached, it is pumped by pump 1² into the softening system pipeline. The volume of used tanks is determined by the frequency and duration of the solid phase removal process, the incoming water initial hardness, the presence of other compounds capable to form a solid phase during softening, the system's capacity, the volume of mechanical filters, etc. Injectors are used to enable the use of low-pressure dosing pumps and can be removed when using other types of pumps.

CONCLUSIONS

The obtained results suggest that effective water softening by treatment with sodium carbonate and sodium hydroxide at low temperatures and without settling is only possible with a 5–10-fold excess of the precipitant dose according to the stoichiometric amount. However, even under such conditions, the use of this method at low temperatures and without settling is low efficiency and can't be recommended for domestic or office use. The use of phosphate anions as a precipitant proved to be more suitable, as they also react with calcium and magnesium ions, forming solid particles. Phosphates react steadily with hardness ions over a wide range of temperatures and pH levels, though their efficiency significantly decreases at temperature below 5 °C.

A negative aspect of phosphates using is the high dispersion of the formed solid phase, which significantly slows down the filtration process. The use of anionic flocculants allows obtaining suspension filtration speed close to filtration speed of distilled water. To maintain the pH of the softened water within the limits defined by regulatory documents, it is proposed to use a mixture of $[(0.75 \div 0.85) \text{Na}_3\text{PO}_4 + (0.25 \div 0.15) \text{NaH}_2\text{PO}_4]$ as the precipitant, with the addition use of necessary amount of flocculant. A technological scheme for water softening has been developed, aimed to be used as a pre-treatment stage in reverse osmosis systems with small and medium capacities.

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