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# The effect of microfiltration pretreatment on the concentration efficiency of kiwifruit juice

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**ABSTRACT**

Kiwi has high antioxidant properties due to high amounts of ascorbic acid (vitamin C) and polyphenolic and flavonoid compounds and is widely consumed in the world. Kiwi juice is very popular due to its easy consumption and longer shelf life than its fruit. Processes such as concentration and clarification require the use of high temperatures or pressures, which cause the reduction of heat-sensitive compounds and the formation of undesirable compounds in the color, taste, and aroma of fruit juices. Nanofiltration is a suitable alternative for thermal processes in fruit juices due to its cost-effectiveness, high efficiency, no need for high temperature and pressure, and simplicity in carrying out the process. In this research, kiwifruit juice (variety of Hayward) was first subjected to a microfiltration process with a polyvinylidene difluoride (PVDF) membrane with a pore size of 0.22 µm and the permeate was entered into the nanofiltration process with a polyamide membrane with a cut-off of 400 Da and the process continued until the concentration of nutritional compounds. After the membrane process, in addition to determining the dominant fouling index by the Hermia model, the physicochemical properties of kiwi fruit juice such as acidity, total soluble solids, turbidity, pH, total polyphenolic and flavonoid compounds, and antioxidant properties were measured. Microfiltration and then performing the nanofiltration process resulted in the concentration of nutritional compounds that by reaching the volume concentration factor equal to 4, polyphenolic compounds increased 7 times (from about 0.0028 mg/100 cc sample to about 0.02 mg/100 cc sample) and flavonoid compounds increased about 10 times (from about 0.164 mg/100 cc sample to about 1.64 mg per 100 cc sample) were concentrated. Also, the fouling study showed that microfiltration as a pre-treatment caused the reduction of suspended particles in kiwifruit juice and delayed the fouling of the nanofiltration.

## 1- Introduction

Kiwi (Actinidia deliciosa) belongs to the family Actinidiaceae [1]. This plant is native to China and is widely cultivated in Chile, New Zealand, and Italy [2]. Currently, China, New Zealand, Greece, Italy, and Iran are the largest kiwi-producing countries in the world [3]. Iran ranks as the sixth-largest exporter of kiwi globally, and this fruit is considered an economically significant commodity compared to other agricultural products with similar cultivation conditions [4]. Kiwi has various cultivars, including Hayward, Abbott, Montty, and Allison, with Hayward being the most popular in the market [5]. Due to its rich nutritional compounds such as vitamin C, polyphenolic compounds, flavonoids, and antioxidant properties, as well as its unique aroma and flavor, kiwi enjoys high consumption worldwide. Research indicates that the vitamin C content in kiwi is about 3 to 5 times higher than that found in citrus fruits [6]. Because of its high levels of vitamin C and polyphenolic compounds, kiwi is referred to as the "king of fruits" [7].

After harvesting, kiwi fruit rapidly softens due to its high moisture content and storage at room temperature, leading to a reduced shelf life and a decline in its nutrient content. Kiwi juice has gained popularity over whole fruits for two main reasons: ease of consumption by consumers and convenience in transport, as well as an extended shelf life compared to whole kiwi fruit [8].

Processes aimed at extending shelf life and enhancing the quality of fruit juices include pasteurization, concentration, and clarification. However, these processes typically require high temperatures, which can lead to thermal degradation of nutrients and the formation of undesirable compounds [9]. The goal of clarification is to eliminate the turbidity in fruit juices; this process reduces the tannin content in the juice, resulting in a decrease in bitter taste. Additionally, concentration increases the shelf life of the juices [10]. On the other hand, clarifying juice before concentrating it prevents large particle burning and improves the quality of the final concentrated product [11].

Membrane processes can serve as suitable alternatives to high-temperature processes due to their ease of operation, lower costs, and lower process temperatures, which result in less nutrient degradation. Microfiltration, ultrafiltration, nanofiltration, and reverse osmosis are examples of membrane processes used for the clarification and concentration of fruit juices [12] and [13]. The advantages of these processes compared to thermal methods include high efficiency and the operation of processes at ambient temperatures, leading to the preservation of nutrients [13].

Due to the very small pore size in nanofiltration, only water and monovalent ions can pass through the membrane surface, while other components, such as proteins and divalent ions, are retained on the membrane. Nanofiltration is a suitable membrane process for concentrating nutrient compounds in the fruit juice industry [14].

In this study, kiwi juice from the Hayward variety, which has the highest cultivated area and marketability among different kiwi varieties, was initially subjected to

microfiltration pretreatment. The permeate obtained from this membrane process then underwent nanofiltration to concentrate nutritional compounds, continuing until the volume concentration factor (VCF) reached 4. Additionally, the physicochemical properties of kiwi juice, including pH, acidity, total soluble solids (TSS), polyphenolic and flavonoid content, and antioxidant activity, were evaluated in both the feed and the concentrated juice. The objective of this research was to apply microfiltration pretreatment to reduce suspended particles in fresh kiwi juice and to prevent severe membrane fouling during the nanofiltration process, followed by performing nanofiltration to concentrate the nutritional compounds in the permeate obtained from this process.

## 2- Materials and Methods

### 1-2- Kiwi Juice Extraction

Hayward kiwi was purchased from a research orchard in the Salman Shahr region of Mazandaran province and stored in a cool, dark place. Once the total soluble solids reached approximately 12 °Brix, the kiwis were washed and manually peeled. The peeled kiwis were then juiced using a three-in-one juicer (Pars Khazar model P610, made in Iran) and filtered. Kiwi juice was stored in one-liter polyethylene terephthalate bottles in a freezer at -20 °C until testing. On the day of testing, the kiwi juice was thawed and passed through two layers of fine mesh to prepare it for the membrane system.

### 2-2- Membrane Process

In this study, kiwi juice was first subjected to microfiltration pretreatment using a polyvinylidene fluoride (PVDF) membrane with a pore size of 0.22 µm, manufactured by Qingfeng, China. The permeate obtained was then directed into a nanofiltration system with a polyamide membrane with a 400 Da cut off, produced by Sharif Water Industry Company, Iran. Throughout all processes, the physicochemical properties of the feed and permeate were evaluated.

The overall layout of the membrane system used is illustrated in Figure 1.

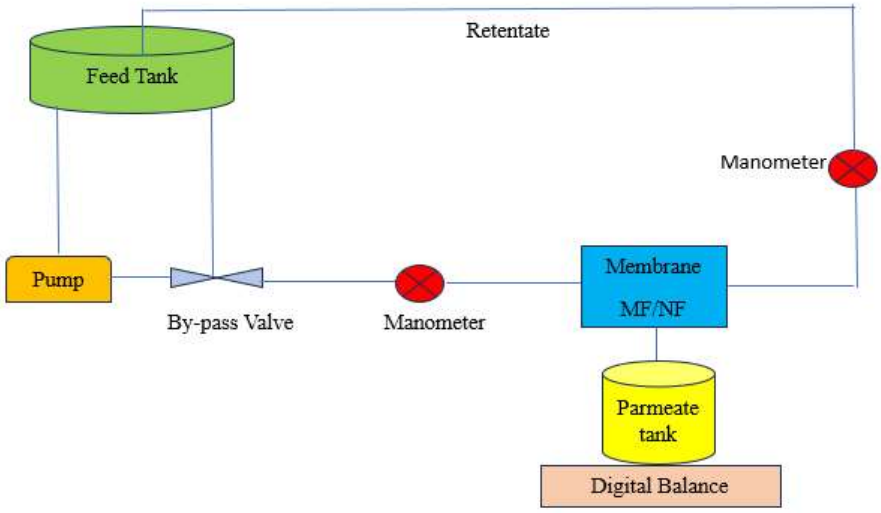



**Fig. 1. schematic of membrane process**

2-3 Measurement of Flux and VCF

To measure flux, the volume of kiwi juice passing through the membrane surface area per unit time was recorded for one hour. For VCF measurement, a specific volume of kiwi juice was introduced into the feed tank, and the process was halted once a specified volume of permeate had been collected. The VCF was then calculated using the formula:

$$\text{VCF} = \frac{feed}{feed - permeate} \times 100$$

2-4 Physicochemical Property Measurements

2-4-1- pH Measurement:

The pH of the samples was measured using a digital pH meter (827 pH Lab-Metrohm, SWISS) at 25 degrees Celsius.

2-4-2- Total Soluble Solids (TSS) Measurement:

The content of soluble solids in the samples was measured using a handheld refractometer (ATAGO, HSR-500, JAPAN), with values reported in °Brix.

2-4-3- Acidity Measurement:

Titratable acidity was measured through titration and reported as citric acid, the predominant acid in kiwi. To do this, 2.5 g of each sample was diluted with 22.5 mL of distilled water, and after adding a few drops of phenolphthalein indicator, it was titrated with 0.1 N sodium hydroxide until a stable pink color was obtained. The acidity of the samples was determined and reported using the following formula:

$$\textbf{Acidity} = \frac{\boldsymbol{X \times 0.0064 \times 100}}{\boldsymbol{2.5}}$$

where X is the amount of NaOH consumed.

2-4-4- Polyphenolic Compounds Measurement:

The polyphenol content was measured using the Folin-Ciocalteau method and expressed as milligrams of gallic acid per 100 mL of sample. Initially, 0.5 mL of the sample was mixed with 1.5 mL of double-distilled water and 0.25 mL of Folin's reagent; then, 0.5 mL of 7.5% calcium carbonate was added. The samples were kept in a dark place for half an hour, and their absorbance was measured at 765 nm using a spectrophotometer (Perkin Elmer, USA, model Lambda 25). To obtain the standard curve, different concentrations of gallic acid were prepared and their absorbances measured at the same wavelength [8].

2-4-5- Flavonoid Compounds Measurement:

The flavonoid content was measured using the aluminum chloride colorimetric method and expressed as milligrams of catechin per 100 mL of sample. To do this, 0.5 mL of each sample was added to 70 µL of 5% sodium nitrite, followed by 0.15 mL of 10% aluminum chloride. After 5 minutes in a 20 °C water bath, 1.3 mL of distilled water and 0.5 mL of 1 M sodium hydroxide were added. The absorbance of the samples was measured at 415 nm using a spectrophotometer (Perkin Elmer, USA, model Lambda 25). For the standard curve, different concentrations of catechin were prepared, and their absorbances were measured at the same wavelength [8].

2-4-6- Antioxidant Activity (DPPH Inhibition Percentage):

The antioxidant activity of the samples was measured using the DPPH method. A solution of 4 mg DPPH in 100 mL of methanol was prepared. To 0.1 mL of each sample and 0.1 mL of methanol, 3 mL of the DPPH solution was added. After placing the samples in a water bath (Serology Water Bath, SHSWB25, IRAN) at 25 degrees Celsius for 20 minutes, their absorbance was measured at 517 nm using a spectrophotometer (Perkin Elmer, USA, model Lambda 25), and the antioxidant activity was calculated using the following formula:

$$DPPH\ (\%inhibition) = [(Abs0 - Abs1)/Abs0] \times 100$$

In this formula, Abs0 is the absorbance of the control (methanol), and Abs1 is the absorbance of the sample [8].

2-4-7- Examination of Fouling Mechanism in Kiwi Juice Clarification

In this study, the equations established according to the Hermia model, used by Satyanarayana et al. in 2023 (Table 1), were employed to investigate the fouling mechanism during the process.

**Table 1. Hermia's model equations [15]**

| S. No | Hermia's Model | Concerning equation |
|---|---|---|
| (a) | Standard pore blocking | $J^{-0.5} = J_0^{-0.5} + k_s t$ |
| (b) | Complete pore blocking | $\ln J^{-1} = \ln J_0^{-1} + k_b t$ |
| (c) | Intermediate pore blocking | $J^{-1} = J_0^{-1} + k_i t$ |
| (d) | Cake filtration | $J^{-2} = J_0^{-2} + k_c t$ |

2-4-8- Calculation of fouling index

In order to calculate the fouling index (IF), the pure water flux was measured before and after the kiwi juice clarification process. Then the fouling index was calculated through the following formula.

$$(IF= 1- \frac{j1}{j0} \times 100)$$

where j1 is the water flux after the process and j0 is the water flux before the process.

2-4-9- Statistical Analysis:

All tests were performed in triplicate, and average values were reported. Statistical analysis of the data was conducted using one-way analysis of variance (ANOVA). Minitab 15 software was used for data comparison using Duncan's multiple range test.

3- Results and Discussion

3-1- Changes in Permeate Flux and VCF in the Microfiltration Process

Hayward variety kiwi juice was introduced into the microfiltration system. Figures 2 and 3 show the permeate flux and VCF in the microfiltration process. As indicated in these figures, there was a significant initial decrease in permeate flux due to concentration polarization, followed by fouling and the formation of a cake layer on the membrane. However, the permeate flux stabilized over time. The VCF, indicating product yield efficiency, increased with time.

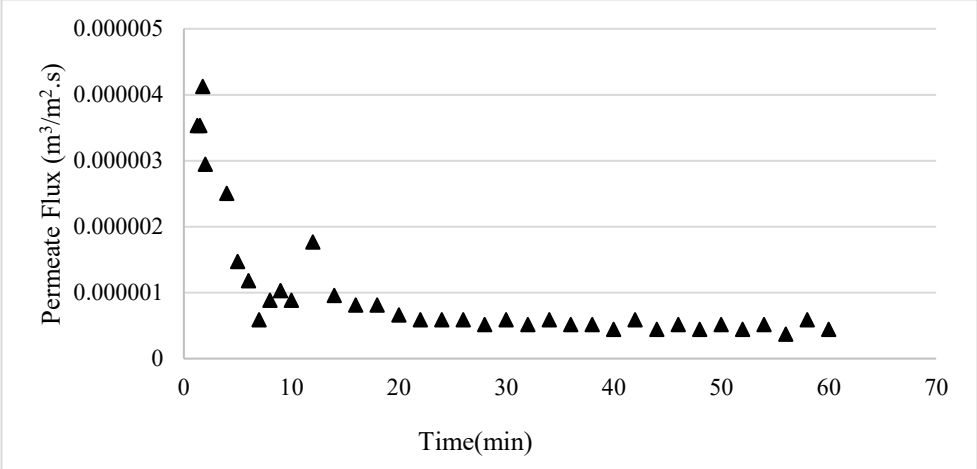



**Fig. 2. Permeate flux of the microfiltration process of kiwifruit juice**

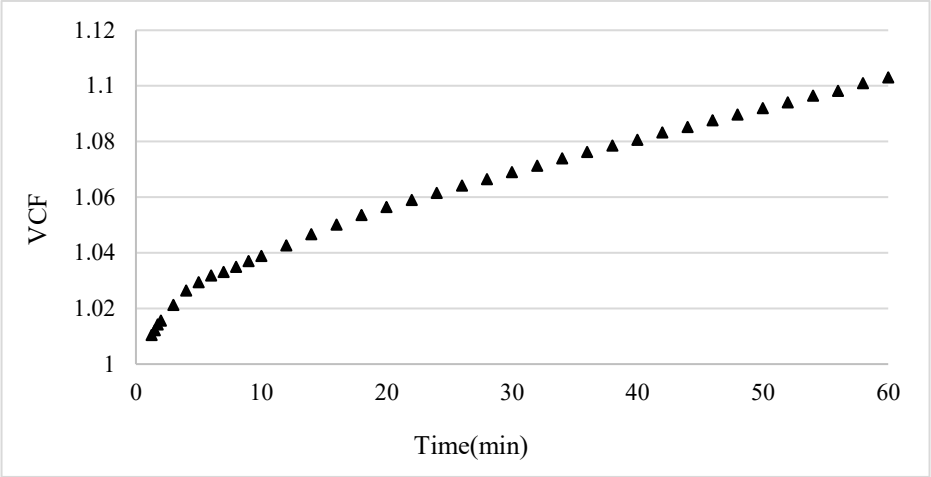
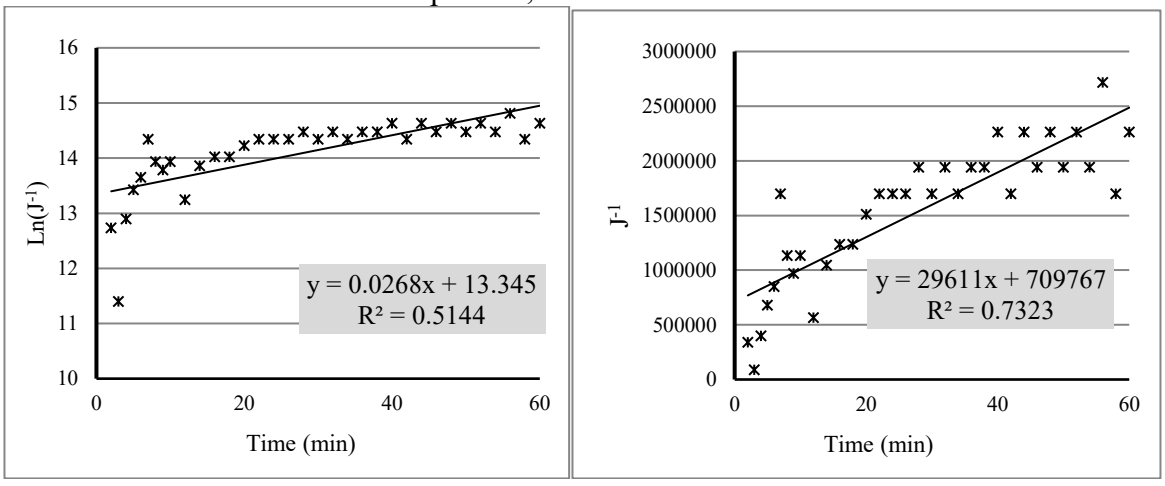



**Fig. 3. changes of VCF during microfiltration process of kiwifruit juice**

3-2- Analysis of the Predominant Fouling Mechanism in Kiwi Juice Clarification by Microfiltration

To analyze the predominant fouling mechanism in the microfiltration process, various Hermia model plots were created based on equations derived from this model, as explained in the subsequent analysis.



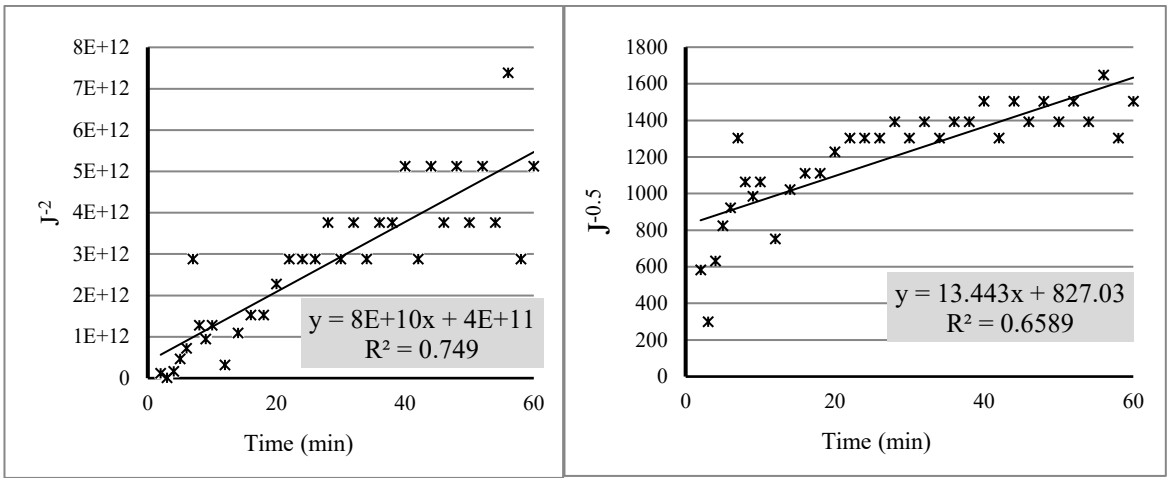



**Fig. 4. The diagrams of Hermia's model during microfiltration of kiwifruit juice**

According to Figure 4, plot $j^{-2}$ shows the highest linearity over time among all the plots. Therefore, cake fouling was identified as the predominant fouling mechanism in the microfiltration of the Hayward variety. Satyanarayana et al., who conducted research on pineapple juice clarification using microfiltration in 2023, also identified cake fouling [16].

3-4- Effects of Microfiltration on the Physicochemical Properties of Kiwi Juice

To assess the effectiveness of the microfiltration process, the physicochemical properties of the permeate from this process were examined.

3-4-1. Effect of Microfiltration on the Soluble Solids Content of Kiwi Juice

The examination of soluble solids content in kiwi juice during the clarification process indicated a significant reduction. This decrease is attributed to the entrapment of molecules such as sugars in the cake layer on the membrane surface (Figure 5). Domingos and colleagues, who worked on microfiltration in passion fruit in 2014, reached a similar conclusion regarding the reduction of soluble solids content in the microfiltration permeate [17].

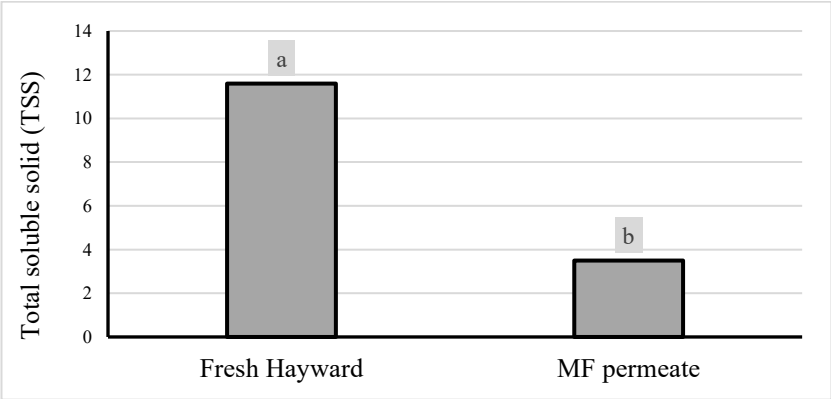


**Fig. 5. The effect of microfiltration on TSS of kiwifruit juice (the same letters means that there is no significant difference at 95%.)**

3-4-2. Effect of Microfiltration on the pH of Kiwi Juice

The study of the effect of microfiltration on the pH of kiwi juice showed a significant increase in pH in the microfiltration permeate compared to fresh kiwi juice (Figure 6). The partial breakdown or entrapment of acidic molecules like vitamin C in the cake layer on the membrane may explain the slight increase in pH in the permeate compared to fresh kiwi juice.

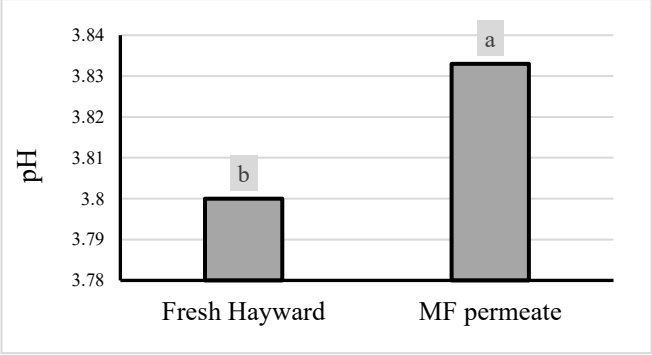
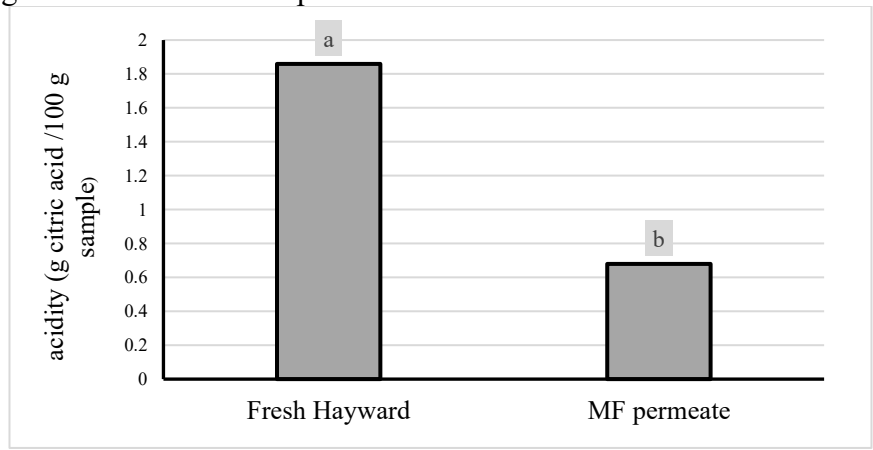



**Fig. 6. The diagram of effect of microfiltration on pH of kiwifruit juice (the same letters means that there is no significant difference at 95%.)**

3-4-3. Effect of Microfiltration on the Acidity of Kiwi Juice

The evaluation of acidity changes in kiwi juice during the microfiltration process showed that acidity variations were in the same direction as pH changes, with a decrease in acidity in the permeate. This reduction is likely due to the breakdown or entrapment of acidic molecules (Figure 7).




**is no Fig. 7. The effect of microfiltration on the acidity of kiwifruit juice (the same letters means that there significant difference at 95%)**

3-4-4. Effect of Microfiltration on Total Polyphenols in Kiwi Juice

Polyphenols are one of the essential nutrients found in kiwi juice. The changes in these compounds during the juice clarification process were examined. Results indicated that polyphenol content in the permeate decreased (Figure 8), attributed to the entrapment of large polyphenol molecules in the membrane's cake layer. Cassano and colleagues, who clarified cactus juice through microfiltration in 2010, reached a similar conclusion [12].

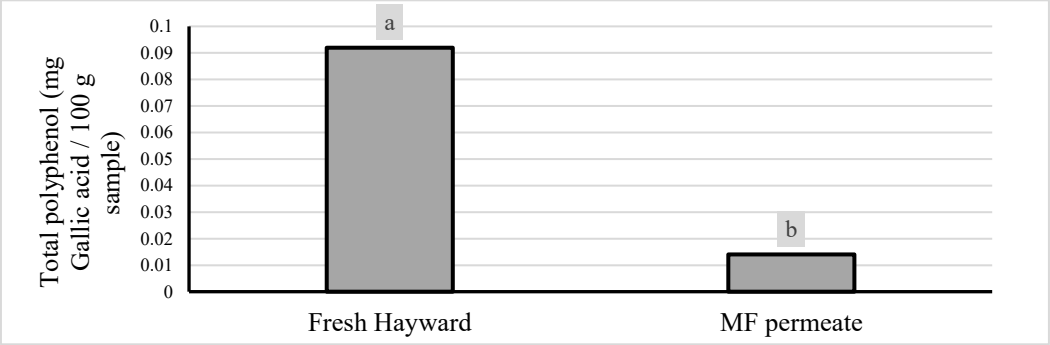



**Fig. 8. The effect of microfiltration on total polyphenol content of kiwifruit juice (the same letters means that there is no significant difference at 95%)**

3-4-5. Effect of Microfiltration on Total Flavonoids in Kiwi Juice

Flavonoids, like polyphenols, are essential nutrients in kiwi juice. Changes in flavonoid content during clarification showed a significant reduction in flavonoids during microfiltration. This decrease is due to their entrapment in the membrane cake layer, their adsorption onto the membrane surface, or degradation due to environmental changes and oxidation (Figure 9). These findings are consistent with Cassano's 2010 study on cactus juice and Mejia's study on pear juice clarification [12] and [18].

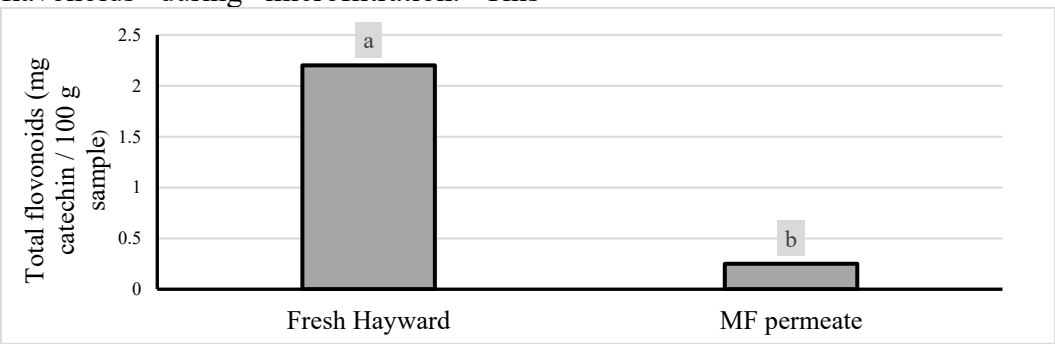



**Fig. 9. The effect of microfiltration on total flavonoids content of kiwifruit juice (the same letters means that there is no significant difference at 95%)**

3-4-6. Effect of Microfiltration on the Antioxidant Properties of Kiwi Juice

The antioxidant properties of kiwi juice mainly result from high levels of ascorbic acid, polyphenols, and flavonoids. Figure 10 shows the antioxidant properties in the

microfiltration permeate and fresh kiwi juice. As indicated, the antioxidant capacity decreased during microfiltration, likely due to vitamin C oxidation and the entrapment of antioxidant molecules in the membrane cake layer. Similar results on the decrease in antioxidant properties in microfiltration permeate were observed in Mejia's study on pear juice clarification [18].

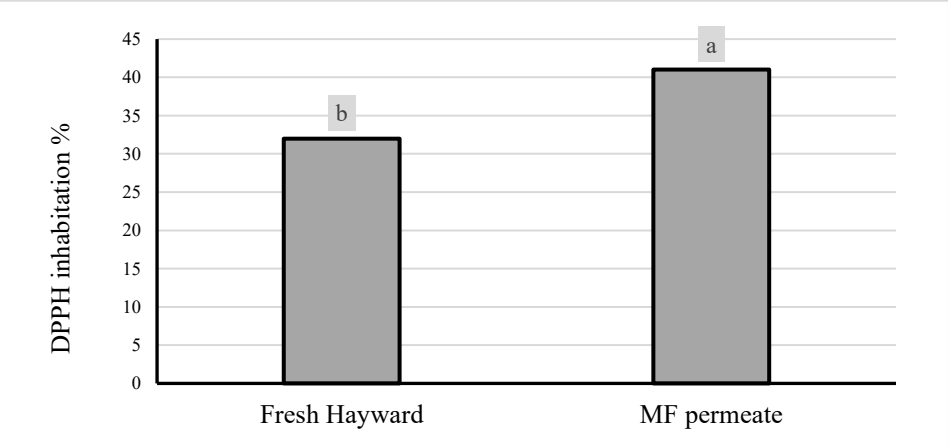



**Fig. 10. The effect of microfiltration on the antioxidant activity of kiwifruit juice (the same letters means that there is no significant difference at 95%)**

3-5- Effect of Microfiltration Pretreatment on VCF in Kiwi Juice Nanofiltration Process

To study the effectiveness of microfiltration pretreatment on kiwi juice concentration efficiency by nanofiltration, concentration was carried out with two feed types: fresh kiwi juice and microfiltered kiwi juice permeate. Figure 11 shows the VCF of kiwi juice following nanofiltration with and without microfiltration pretreatment. Microfiltration pretreatment produced clarified kiwi juice by removing large molecules and entrapping them in the membrane cake layer. When this clarified kiwi juice was used in nanofiltration, less fouling occurred, and VCF was higher than for fresh kiwi juice containing more suspended particles.

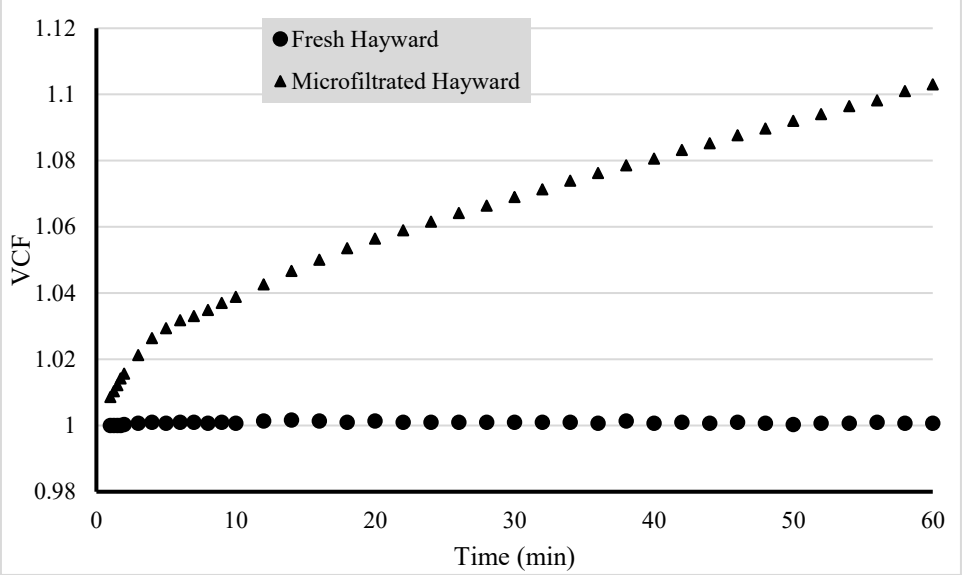



**Fig. 11. The VCF change of nanofiltration process of kiwifruit juice with and without microfiltration pretreatment**

3-6- Evaluation of Fouling Index in Kiwi Juice Clarification Process by Microfiltration

The fouling index for the microfiltration process was calculated after testing and assessing permeate flux changes and VCF levels. As shown in Figure 12, due to the high content of suspended particles, the fouling index for fresh kiwi juice was 100, indicating complete membrane fouling. The fouling index in the microfiltration permeate was lower than that of fresh kiwi juice, due to partial clarification during microfiltration.

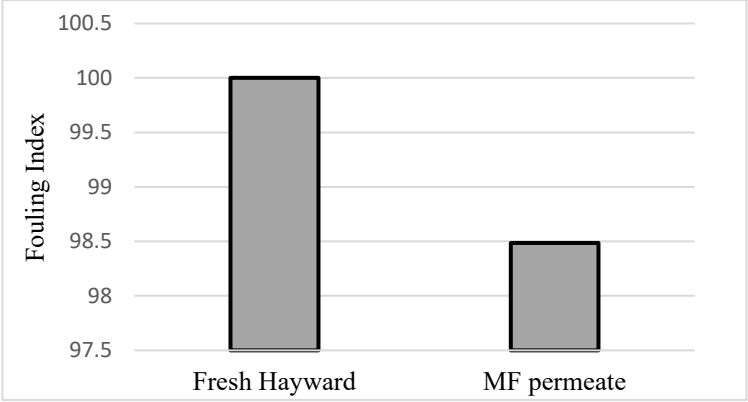



**Fig. 12. Fouling index of the microfiltration process**

3-7- Dominant Fouling Mechanism in Nanofiltration Process

To study the dominant fouling mechanism in the nanofiltration process, two feeds (fresh kiwi juice and microfiltration permeate) were tested, and various plots

based on Hermia's model were generated for further analysis.

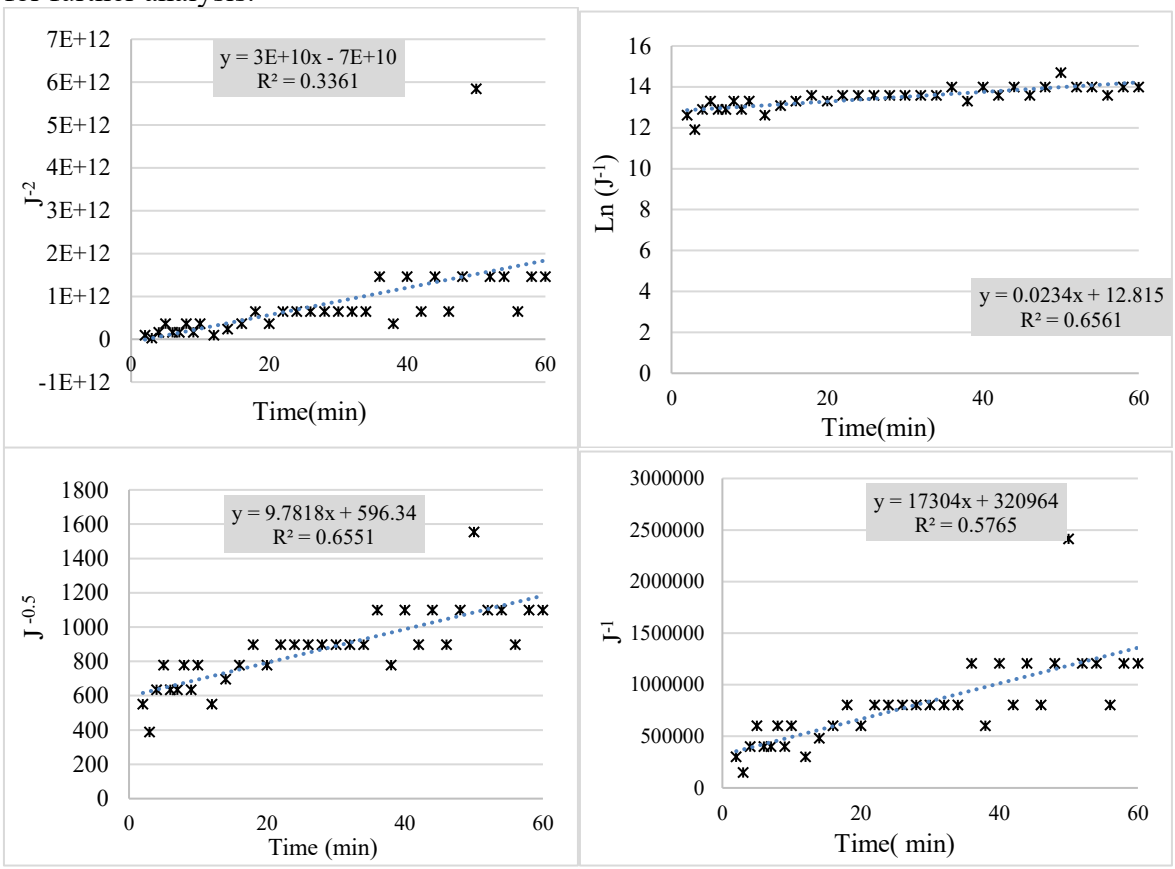



**Fig. 13. The diagrams of Hermia's model during nanofiltration of kiwifruit juice with fresh fruit feed**

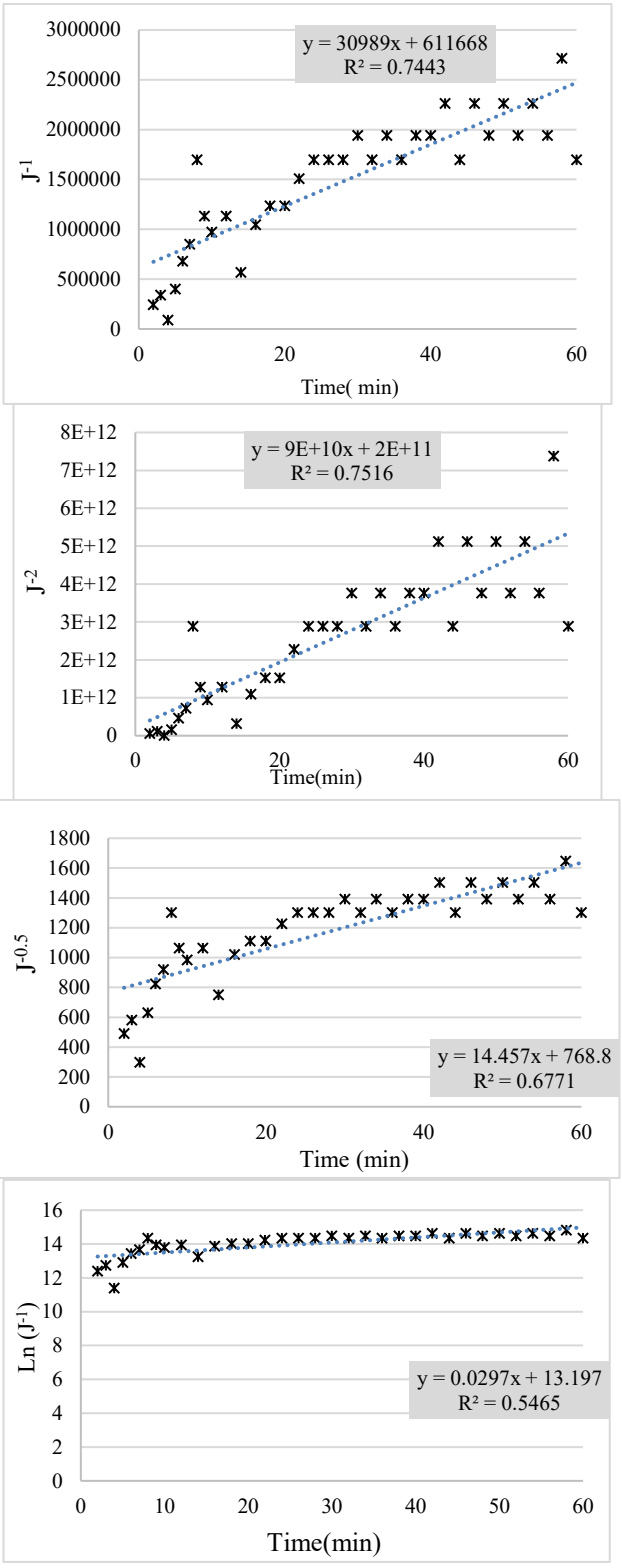



**Fig. 14. The diagrams of Hermia's model during nanofiltration of kiwifruit juice with permeate of microfiltration feed**

According to Figures 13 and 14, the Ln$j^{-1}$ plot and $j^{-2}$ plot showed the highest linearity over time among other plots respectively. Therefore, complete pore blocking was the dominant fouling mechanism during the nanofiltration of fresh kiwi juice, whereas cake fouling was the dominant mechanism in nanofiltration of microfiltration permeate. Liu and colleagues in 2021 identified cake fouling as the dominant fouling mechanism in beet juice clarification using Hermia's model [19].

3-8. Effect of Nanofiltration with Microfiltration Pretreatment on the Physicochemical Properties of Kiwi Juice

After microfiltration, the resulting permeate entered the nanofiltration process and was concentrated to a VCF of 4. The physicochemical properties of the nanofiltration retentate were then measured.

Figure 15 shows the physicochemical properties of kiwi juice concentrated by nanofiltration after microfiltration pretreatment. Flavonoid and polyphenol contents in the nanofiltration retentate were higher than in fresh kiwi juice. Polyphenolic compounds were concentrated approximately seven times, while flavonoids were concentrated around ten times. Due to the microfiltration process, suspended particles and large molecules in fresh kiwi juice were partially removed, reducing severe fouling in the membrane during nanofiltration and allowing for nutrient concentration as the nanofiltration process continued. Though significant quantities of phenolic and flavonoid compounds may have been trapped in the pre-filtration cake layer, nanofiltration compensated for this reduction, significantly increasing the concentration of these compounds in the final concentrated product. The results also showed minimal changes in pH and TSS after membrane concentration, with antioxidant properties remaining stable. This stability is due to the balancing effect of phenolic compound concentration and vitamin C degradation during the membrane process, as well as vitamin C entrapment in the cake layer on the membrane.

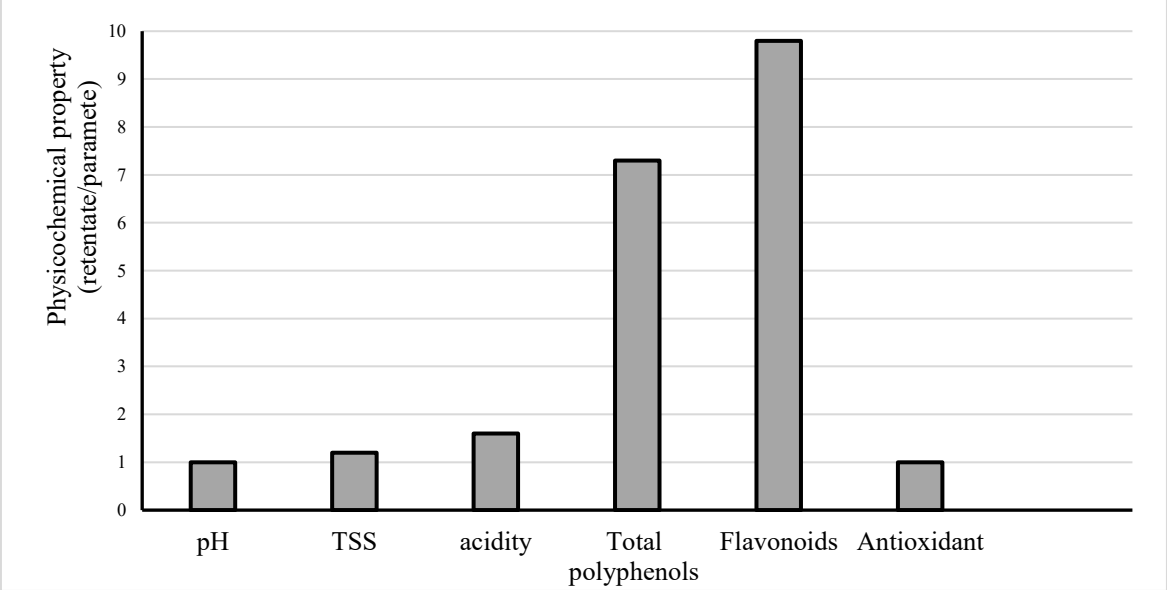



**Fig. 15. The effect of nanofiltration with microfiltration pretreatment on the physicochemical properties of kiwifruit juice**

## 4- Overall Conclusion

Microfiltration pretreatment reduced suspended particles and large molecules in fresh kiwi juice, allowing the use of a nanofiltration membrane with extremely small pores. Consequently, membrane fouling occurred less frequently and more slowly than when fresh kiwi juice was used directly in nanofiltration, resulting in higher product yield. Continuing nanofiltration to reach a minimum VCF of 4 effectively concentrated nutrients in kiwi juice. Therefore, pretreatment of kiwi juice with microfiltration, followed by nanofiltration to a VCF of 4, offers a feasible and effective method for concentrating kiwi juice.

## Data Availability

The data used to support the finding of this study are available from the corresponding author upon request.

## Conflict Of Interest

The authors have no conflicts interest to report.

## Funding Statemant

The researchers did not receive any specific grant from funding agencies the public, commercial or not-for-profit sectors.

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مقاله علمی_پژوهشی

# اثر پیش تیمار ریزپالایش بر راندمان تغلیظ آب کیوی

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**چکیده**

کیوی به دلیل داشتن مقادیر بالای اسید آسکوربیک (ویتامین C) و همچنین ترکیبات پلی‌فنلی و فلاونوئیدی دارای خاصیت آنتی‌اکسیدانی بالایی بوده و مصرف گسترده‌ای در جهان دارد. آب کیوی به جهت مصرف آسان و مدت زمان نگهداری بالاتر نسبت به میوه آن محبوبیت زیادی دارد. فرایندهایی مانند تغلیظ و شفاف‌سازی نیازمند استفاده از دما و یا فشارهای بالا می‌باشند که به دنبال آن سبب کاهش ترکیبات حساس به حرارت و تشکیل ترکیبات نامطلوب در رنگ، طعم و عطر آبمیوه‌ها می‌شوند. فرایند نانوفیلتراسیون به دلیل مقرون به صرفه بودن، راندمان بالا، عدم نیاز به دما و فشارهای بالا و سادگی در انجام فرایند جایگزین مناسبی برای فرایندهای حرارتی در آبمیوه‌ها می‌باشد. در این تحقیق آب کیوی واریته هایوارد ابتدا تحت پیش فرایند ریزپالایش با غشاء پلی وینیلیدین دی فلوئورید (PVDF) با اندازه منفذ ۰/۲۲ µm قرار گرفت و تراوه حاصل از آن وارد فرایند نانوفیلتراسیون با غشاء پلی آمید با کات آف ۴۰۰ دالتون گردید و فرایند مذکور تا زمان تغلیظ ترکیبات تغذیه‌ای ادامه یافت. پس از انجام فرایند غشایی، علاوه بر تعیین شاخص غالب گرفتگی توسط مدل هرمیا، خواص فیزیکوشیمیایی آب کیوی مانند اسیدیته، مواد جامد محلول، کدورت، pH، ترکیبات پلی‌فنلی و فلاونوئیدی کل و خاصیت پاداکسایندگی در هر مرحله سنجیده شده و مشخص گردید پیش‌تیمار ریزپالایش و سپس انجام فرایند نانوفیلتراسیون سبب تغلیظ ترکیبات تغذیه‌ای گردید؛ به نحوی‌که با رسیدن به فاکتور غلظت حجمی برابر با ۴، ترکیبات پلی‌فنلی ۷ برابر (از حدود ۰.۰۰۲۸ میلی‌گرم در ۱۰۰ سی‌سی نمونه به حدود ۰.۰۲ میلی‌گرم در ۱۰۰ سی‌سی نمونه) و ترکیبات فلاونوئیدی حدود ۱۰ برابر (از حدود ۰.۱۶۴ میلی‌گرم در ۱۰۰ سی‌سی نمونه به حدود ۱.۶۴ میلی‌گرم در ۱۰۰ سی‌سی نمونه) تغلیظ گردیدند. همچنین مطالعه گرفتگی نشان داد که ریزپالایش به عنوان پیش تیمار، سبب کاهش ذرات معلق در آب کیوی شد و گرفتگی در غشای نانوفیلتراسیون را به تعویق انداخت.