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# DESIGN AND PROTOTYPE PRODUCTION OF UNINTERRUPTED CLEAR FRUIT JUICE PRODUCTION LINE WITH TANDEM FEED ULTRAFILTRATION METHOD

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## ABSTRACT

In the fruit juice industry, ultrafiltration (UF) technology has gained great importance in the production of clear fruit juice in recent years. In this study, by determining the clogging period of the membrane with turbid product inlet and clear product outlet pressure in the UF membrane, the limit impurity value was determined and turbid product was fed accordingly. In this process, the dark product coming out of the membrane was stored in one of the tandem tanks and fed to an external decanter from this tank after a certain period of time. The clear product from the decanter was sent to the clear product tank feeding UF, which was still in production. Thus, the CIP process, which is mandatory in case of clogging of the membranes, was postponed and the diafiltration process was carried out externally, ensuring internal uninterrupted production. In addition, it was aimed to prevent fouling by surface modification with various polyelectrolytes for the membranes used in these processes. Clarity, iodine, alcohol, pH tests, brix measurement, SEM and contact angle analyses were performed on coated and uncoated membranes under 3 and 5 bar pressure. By delaying fouling, CIP process time was reduced and production time was increased. Thus, production efficiency was increased and CIP chemical costs and water consumption were reduced by shortening the CIP time and number. In addition, water consumption during the diafiltration process is prevented. The high efficiency and low cost of the developed system makes UF systems preferable and accessible.

**Keywords:** Fruit Juice, Clarification, Ultrafiltration

## 1. INTRODUCTION

Fruit juices are nutritious beverages obtained mechanically from healthy, ripe, fresh and clean fruits, showing the typical characteristics of the fruit from which they are obtained, such as color, taste and smell, not fermented but fermentable and physically resistant. The transformation of fruit into fruit juice varies according to the fruit concerned, the processing technology, the quality of the end product and consumer demand. Depending on the natural characteristics of the fruit and consumption habits, some fruits (apple, grape, sour cherry, pomegranate) are processed in a clearer way and some fruits (peach, apricot) are processed in a cloudy way. Ultrafiltration (UF) is an increasingly widespread membrane filtration technology in the fruit juice industry for one-step clarification of the product and obtaining high quality juice Katibi et al. (2023). Conventional clarification processes are typically multi-stage, requiring steps such as enzymatic depectination, followed by precipitation with clarifying agents such as gelatin-bentonite and then kieselgur filtration, which is both time-consuming and labour-intensive Celikten et al. (2022). Ultrafiltration can clarify juice by separating suspended particles, pectins and turbidity-causing colloids in a single step, thus largely eliminating the need for centrifugation, auxiliary enzymes or clarification chemicals. This can shorten processing times by more than 80 per cent compared to the conventional method and provides a more economical production by increasing product yield. The UF process operated at low temperature preserves the nutritional value and flavour of the juice better than heat treatments, and the absence of additional chemicals leaves no undesirable residues in the product Katibi et al. (2023). Today, ultrafiltration is successfully applied in the clear production of many fruit juices such as apple, pomegranate, grape, citrus, pineapple, guava, dragon fruit and banana juice. In addition, since UF systems are modular and compact, they can be easily integrated into juice plants and offer an environmentally friendly alternative that contributes to sustainable production targets Celikten et al. (2022).

Studies conducted in the last 5 years reveal the significant effects of ultrafiltration technology on productivity and product quality in fruit juice production. One of the most important parameters is membrane fouling and methods to prevent it. For example, in a study on banana juice, pre-treatment with 0.1-0.5% pectinase enzyme before UF reduced the formation of gel layer on the membrane surface by degrading pectin and reduced the fouling effect. The viscosity of banana juice treated with pectinase decreased by more than 50% and the average permeate flux obtained during ultrafiltration increased 1.65 times to 24 kg/m$^2$-hr compared to the non-enzyme treated sample Yee et al. (2021). Similarly, when undesirable components such as tannins were removed from cashew apple juice sample by pre-precipitation by centrifugation, followed by membrane filtration, turbidity removal up to 97% and high permeate flux were obtained Abdullah et al. (2022). These findings suggest that pretreatment steps play a critical role in improving UF process efficiency. Indeed, the literature suggests the application of hybrid approaches such as enzymatic treatment of feedstock, pre-filtration or clarification to delay membrane fouling Jönsson et al. (2013).

The type of membrane and pore size (MWCO) used in the ultrafiltration process also greatly affect juice yield and quality. Recent studies have shown that the choice of an optimum membrane cut-off threshold maximizes flux by reducing fouling. In an experiment with red dragon fruit juice, three polymeric UF membranes with different MWCO values were compared and the highest permeate flux was obtained with the 10 kDa porous membrane at 300 kPa transmembrane pressure. It has been reported that larger or smaller MWCO values, if incompatible with the colloid size distribution, reduce performance due to gel layer formation and pore clogging Le et al. (2021). In terms of membrane material, most research in the last five years has focused on polymeric membranes (especially polysulfone (PSF) and polyether sulfone (PES) based membranes) because these membranes offer high selectivity, mechanical strength and chemical resistance in food applications. Indeed, PSF/PES membranes are the most widely used membrane types for juice clarification, successfully retaining tannins, proteins, starch, yeast and colloids and providing clarity up to 99% Katibi et al. (2023). On the other hand, ceramic membranes have also attracted interest in recent studies. Ceramic membranes are more resistant to fouling and cleaning processes and have been used in some pilot applications for pre-treatment of fruit juice by microfiltration or clarification by UF. For example, it was reported that a permeate flux of 273 L/m$^2$ per hour was achieved and >99% turbidity removal was achieved in watermelon and kiwi juice filtration with a metakaolin-based ceramic membrane Hubadillah et al. (2019). However, due to their high cost, the use of ceramic membranes is limited in the industry and polymeric membranes are preferred Katibi et al. (2023). Improving the performance of membranes by surface modification has been an important research topic in theses and articles published in recent years. For example, hydrophilicity was increased by adding titanium dioxide ($TiO_2$) and alumina ($Al_2O_3$) nanoparticles to new generation nanocomposite UF membranes, resulting in a high flux of 44.6 L/m$^2$-hr in apple juice ultrafiltration and lower turbidity and higher phenolic content compared to commercial clear apple juice Severcan et al. (2020). It has also been reported that the flux can be increased by ~80% and membrane fouling is significantly reduced with chitosan-polypyrrole doped antifouling membranes developed for sugarcane juice Akhtar et al. (2020). In addition, some innovative research has experimented with external field applications: For example, up to 99% turbidity removal and higher permeate flux were achieved in UF/MF filtration of pomegranate juice by applying laser radiation Salehinia et al. (2021). Similarly, magnetic field application was shown to increase pomegranate juice filtration efficiency by reducing membrane fouling Zarouk et al. (2022). All these studies reveal that UF technology has made significant progress in process optimization (optimization of parameters such as pressure, flow rate, temperature, etc.) and membrane development in recent years.

## 2. MATERIAL AND METHOD

Within the scope of this study, firstly, the effect of coating on 5 kDa and 10 kDa membranes was studied at 5 bar pressure using both coated and uncoated membranes and apple juice. Polyallylamine hydrochloride (PAH) solution was used as the coating solution. The resulting juice samples were stored at -18°C and the membranes used were kept in sample containers with water.

As a next step, filtration studies were carried out with apple juice at 3 bar using coated and uncoated membranes at 5 kDa and 10 kDa membranes, different from 5 bar, to study the effect of pressure on separation performance and membrane fouling. Coated membranes were prepared by soaking the membranes in the coating solution for about 16 hours. The retentate and permeate samples obtained from these studies and the membranes used were stored for further analysis.

Clarity test, iodine test, alcohol test, pH test, brix measurement, SEM and contact angle analysis were performed on coated and uncoated membranes obtained under 3 and 5 bar pressure. Clarity test was performed by transmittance at 625 nm with a spectrophotometer, iodine and alcohol tests were performed chemically, pH test was measured with a pH meter and brix measurement was performed gravimetrically in an oven.

**Iodine test:** The iodine test is performed to determine the presence of starch, which causes turbidity in the juice. Normally, the enzyme added to break down pectin also breaks down starch. In the iodine test performed on the samples obtained as a result of filtration studies, 10% iodine solution was used. While preparing the iodine solution, first 0.1 g iodine is dissolved in 2.0 ml ethyl alcohol and 2.0 g potassium iodide is added and dissolved with a small amount of distilled water. Finally, the solution is made up to 100 ml with distilled water. Put 10 ml of the juice permeate sample to be tested for iodine into a test tube. A few drops (approximately 1 ml) are added with a pipette by tilting the test tube so that it is not directly into the fruit juice solution. The iodine solution allowed to flow through the wall of the test tube is allowed to mix with the juice solution and color formation is observed. In color formation, blue color indicates the presence of undegraded starch, lilac color indicates that the degradation of starch is in the initial stage, brown indicates that dextrins have started to form from starch, and red color indicates that starch has been degraded into dextrins. In this case, red color was sought in fruit juice samples.

**Alcohol test:** The alcohol test is performed to check whether the pectin degradation by enzymatic action is sufficient in fruit juice. For the alcohol test, a mixture prepared using 96% ethyl alcohol and 5% HCL solution is used. This mixture contains 95 ml of 96% ethyl alcohol and 5 ml of 5% HCL solution. For the alcohol test, 5 ml of the juice permeate sample is taken into the test tube and 10 ml of the ethyl alcohol-HCL mixture is added, the tube is shaken and the tube is left alone. If pectin-induced sediment appears in the tube within 1 minute, pectin enzymation is incomplete. However, since enzymated fruit juice is supplied for use in laboratory studies, sediment formation in the tubes is not expected in the alcohol test.

**Brix measurement:** Brix (dry matter) of permeate and retentate samples was measured gravimetrically. For this, petri dishes dried in an oven at 105°C for 3 hours and cooled in a desiccator for half an hour were weighed and tared. A certain amount of fruit juice sample was placed in the petri dishes and after weighing the sample amount, the petri dish was dried in an oven at 105°C for about 4 hours. The dried samples are cooled in a desiccator for half an hour. The cooled petri dishes are weighed again and the amount of moisture (water) removed from the juice sample is calculated and the dry matter is calculated.

**Calculation of the fouling index:** To calculate the fouling indices of the membranes, pure water is passed through clean membranes and the time required to collect 10 ml of permeate sample is determined. Then a filtration study is performed with these membranes for 15 minutes. Then, pure water is passed through the same membrane again and the time required to collect 10 ml permeate sample is determined and the fouling index is calculated with the formula below.

Fouling index = (1-(tf/ti))/t*100

**ti:** Time taken to collect specific permeate sample with clean membrane

**tf:** Time taken to collect specific permeate sample with fouled membrane

**t:** 15 min, time required for filtration of the sample of interest through the membrane

## 3. RESULTS

**Table 1**

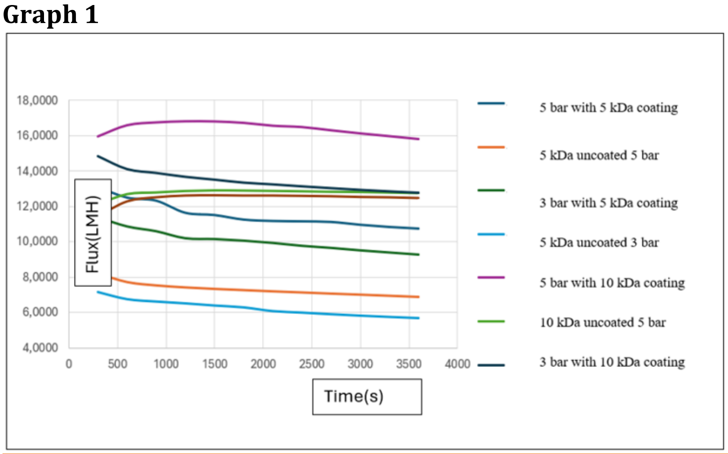
**Table 1 Analysis Results**

| Sample Name | pH | Brix° (%) | Iodine (color) | Alcohol (sedimentation) | Clarity (T625) |
|---|---|---|---|---|---|
| Feed Sample | 3,71 | 11,2 | n.d. (red) | n.d. | 57,4 |
| 5 Bar Permeate With 5 Kda Coating | 3,9 | 9,1 | n.d. (red) | n.d. | 117,3 |
| 5 Bar Retentate With 5 Kda Coating | 3,66 | 10,7 | n.d. (red) | n.d. | 27,1 |
| 3 Bar Permeate With 5 Kda Coating | 3,84 | 8,5 | n.d. (red) | n.d. | 116,2 |

| | | | | | |
|---|---|---|---|---|---|
| 3 Bar Permeate With 5 Kda Coating | 3,59 | 10,2 | n.d. (red) | n.d. | 38 |
| 5 Kda Uncoated 5 Bar Permeate | 3,83 | 8,6 | n.d. (red) | n.d. | 116,7 |
| 5 Kda Uncoated 5 Bar Retentate | 3,62 | 10,3 | n.d. (red) | n.d. | 33,6 |
| 5 Kda Uncoated 3 Bar Permeate | 3,78 | 7,9 | n.d. (red) | n.d. | 115,6 |
| 5 Kda Uncoated 3 Bar Retentat | 3,53 | 9,8 | n.d. (red) | n.d. | 41,9 |
| 5 Bar Permeate With 10 Kda Coating | 3,94 | 10,4 | n.d. (red) | n.d. | 108,7 |
| 10 Kda Coated 5 Bar Retentate | 3,48 | 11,4 | n.d. (red) | n.d. | 32,3 |
| 3 Bar Permeate With 10 Kda Coating | 3,86 | 9,8 | n.d. (red) | n.d. | 106,9 |
| 3 Bar Retentate With 10 Kda Coating | 3,52 | 11 | n.d. (red) | n.d. | 41,8 |
| 10 Kda Uncoated 5 Bar Permeate | 3,82 | 10 | n.d. (red) | n.d. | 106,3 |
| 10 Kda Uncoated 5 Bar Retentate | 3,61 | 11,2 | n.d. (red) | n.d. | 38,6 |
| 10 Kda Uncoated 3 Bar Permeate | 3,78 | 9,2 | n.d. (red) | n.d. | 105,2 |
| 10 Kda Uncoated 3 Bar Retentate | 3,64 | 10,7 | n.d. (red) | n.d. | 47,5 |

*** n.d.:** not detected

**Graph 1**




**Graph 1** Flux Comparisons

When coated and uncoated membranes are considered separately in the relevant graph, it is seen that the flux increases as the pressure increases. When a general comparison is made, it is seen that the coated membranes have a significant effect on the flux, even the 3 bar flux results in coated membranes are higher than the 5 bar flux results in uncoated membranes. SEM analysis results show that 10 kDa uncoated 5 bar working membrane is dirtier than 10 kDa coated 3 bar working membrane and 5 kDa uncoated 5 bar working membrane is dirtier than 5 kDa coated 3 bar working membrane, which both explains and confirms the difference between the flux results.

- The results in the table show that the pH of the retentate and permeate samples do not differ much from each other and from the feed solution, but there is a slight decrease in the retentate samples and a slight increase in the permeate samples.
- The Brix° (dry matter) results show that the Brix° values of the permeate samples are lower than the Brix° values of the retentate samples. This is based on the retention of large molecules in the feed solution by the membrane. Since the amount of material passing through the membrane increases with increasing pressure, it is seen that the Brix° value increases with increasing pressure in permeate samples. Brix° values of permeate samples obtained with 5 kDa membrane are lower than Brix° values of permeate samples obtained with 10 kDa membrane. This is due to the fact that 5 kDa membrane has smaller pores than 10 kDa membrane and 5 kDa membrane retains more particles. When the permeate samples of coated and uncoated membranes are compared, it is seen that the Brix° values of the permeate samples obtained with coated membranes are higher. This is due to the fact that the membrane surface is fouled for a longer time due to the coating and more amount of material passes through the membrane in the same period of time.
- Looking at the clarity values, it is seen that the clarity results of the permeate samples are generally close to each other. The slight difference between the clarity of 10 kDa and 5 kDa membrane studies of permeate samples can be interpreted as some particles that may cause turbidity may be retained with a membrane with smaller pores such as 5 kDa compared to 10 kDa membrane. Looking at the clarity values of the retentate samples, it is seen that the clarity is lower than the clarity of the feed solution. This is due to the retention of the particles causing turbidity by the membrane.
- When the results of the alcohol test were analyzed, no sediment was formed in the samples, indicating that the enzymation of pectin was completed.
- When the iodine test results are examined, the red color observed in the samples shows that there is no starch in both the feed solution and the samples obtained from the membran studies.

**Figure 1**

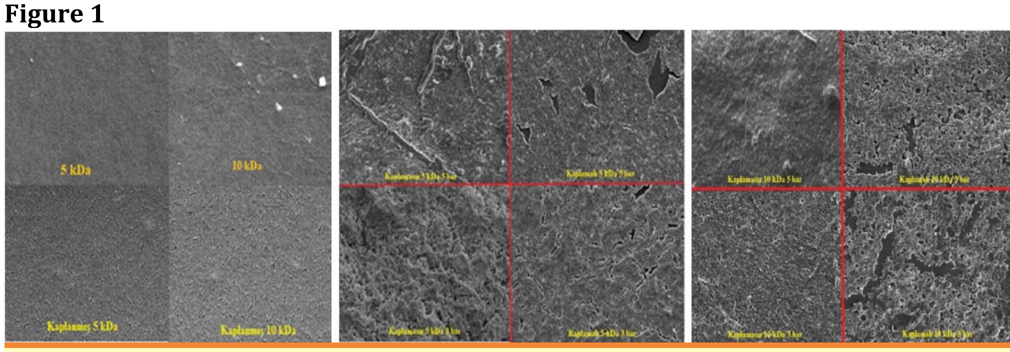



**Figure 1** SEM Analysis Results

SEM analysis results show that the surface of the 5 kDa membrane is generally more fouled than the 10 kDa membrane. This is since the 5 kDa membrane holds more particles because it has smaller pores. When we look at the comparison of coated and uncoated membranes, it is seen that while the surface of the uncoated membranes is completely fouled, the coated membrane surfaces are less fouled and there are gaps in places (i.e. those areas are not fouled). This situation is interpreted as the fouling tendency of the surface decreases because of the coating applied on the membrane surface.

**Table 2**

**Table 2 Contact Angle Results of Clean Membranes**

| Sample | 5 KDA | 5 KDA Coating | 10 KDA | 10 KDA Coating |
|---|---|---|---|---|
| Contact Angle (°) | 58,32 | 39,01 | 60,26 | 40,97 |

**Table 3**

**Table 3 Contact Angle Results of Dirty Membranes**

| Sample | 5 kDa membrane TA (°) | 10 kDa membrane TA (°) |
|---|---|---|
| 5 bar Coating | 56,25 | 51,26 |
| 5 bar Uncoated | 64,97 | 60,91 |
| 3 bar Coating | 49,70 | 47,87 |
| 3 bar Uncoated | 58,32 | 53,42 |

When the contact angle results are analyzed, it is seen that the surface coating of the membrane decreases the contact angle. The decrease in the contact angle is interpreted as more water absorption of the membrane surface, i.e. increased hydrophilicity, i.e. increased flux. Looking at the contact angles as a result of fruit juice studies, it is seen that the contact angles of the 3 bar studies are lower than the contact angles of the 5 bar studies, i.e. the membranes were less fouled in the 3 bar study. When the contact angles of coated and uncoated membranes are compared, it is seen that the contact angles of uncoated membranes are higher than the contact angles of coated membranes under the same conditions, which means that the surface is more fouled.

**Table 4**

**Table 4 Fouling Indices**

| Sample name | tf (s) | ti (s) | t (s) | Fouling index, % |
|---|---|---|---|---|
| 5 bar with 5 kDa coating | 13 | 6 | 15 | 3,59 |
| 5 kDa uncoated 5 bar | 21 | 6 | 15 | 4,76 |
| 3 bar with 5 kDa coating | 11 | 6 | 15 | 3,03 |
| 5 kDa uncoated 3 bar | 18 | 6 | 15 | 4,44 |
| 10 kDa coated 5 bar | 6 | 3 | 15 | 3,33 |
| 10 kDa uncoated 5 bar | 9 | 3 | 15 | 4,44 |
| 3 bar with 10 kDa coating | 5 | 3 | 15 | 2,67 |
| 10 kDa uncoated 3 bar | 7 | 3 | 15 | 3,81 |

When the fouling index results are examined, it is seen that the fouling index, i.e. the fouling tendency, increases as the pressure increases. When coated and uncoated membranes are examined, it is seen that the fouling indexes of coated membranes are lower, that is, their fouling tendency is lower than uncoated membranes. Membranes with fouling index values lower than 3% and 3% should be cleaned every few months, while membranes with fouling index values between 3-5% should be cleaned more frequently than membranes with fouling index values of 3% and lower.

## 4. CONCLUSION

Within the scope of this study, it is aimed for the production of clear fruit juice for the beverage sector; It can prevent fouling, which is an important problem due to the very small size and compressible nature of the particles that are desired to be separated in the filtration process, and does not require diafiltration (re-feeding the solid output to the membrane by re-diluting it with water), which is done in existing systems in order to reuse the solid output, A new system design and development study will be carried out to design and develop a new system with high added value, which has high production efficiency and periodicity, low CIP number requirement, low CIP chemicals and operating costs, and which can be one step ahead of both conventional systems widely used in our country and advanced imported systems. Preventing fouling, which is an important problem, especially in membrane systems, will cause both the treatment performance of the relevant process to be stable for a longer period and the time, chemical, etc. costs to be used for cleaning will be reduced. For this reason, it is important to delay membrane fouling in all industrial processes where membranes are used regardless of the application type, and the fouling resistance of the membranes used in the membrane processes (especially UF process) in my current project will be improved.

In conclusion, continuous clear juice production by ultrafiltration is a very promising approach in the light of recent academic studies and theses. UF systems have become a strong alternative to traditional methods by providing high quality and stable clear juice in a shorter time.

## CONFLICT OF INTERESTS

None.

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