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The Evaluation of Milk Nutrient Content Filtrated by Polyethersulfone Ultrafiltration Membrane with Different Polymer Concentration and Time Filtration

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Abstract *- The aim of this study was to determine the effect of polyethersulfone (PES) polymer concentration and filtration time toward the membrane performance (flux) and milk nutrient content (water content, fat and protein). Ultrafiltration membrane was made using PES polymer and polyethylene glycol (PEG) as additive. This research used Split Plot in Time design with five repetitions. The main plot was filtration time and the subplot was level of polymer concentration. The data was analyzed using analysis of variance (ANOVA) and continued by Duncan Test in 5% level if there was a significant effect. The result showed that there was no interaction effect (P>0,05) between polymer concentration and time filtration to membrane flux and milk nutrient content. The difference of polymer concentration affect membrane flux and the milk water content (P<0,05), while the filtration time only affect the flux (P<0,05). The difference of polymer concentration and time filtration did not give significant effect (P>0,05) toward fat and protein content. Water content in milk filtrated by M1 and M3 membrane increased significantly, whereas water content in milk filtrated by M2 membrane decreased significantly. Fat content in milk filtrated by M1, M2 and M3 membrane were decreased. Milk protein content filtrated by M1 and M2 membrane tend to increase, while milk protein content filtrated by M3 membrane tend to decrease. It could be concluded that M2 membrane that containing of 15% PES and 5% PEG was the best membrane for milk filtration with 5 hours time of filtration.*

Keywords *— flux, membrane, milk nutrient, polyethersulfone, ultrafiltation.*

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I. Introduction

Dairy farm is one of business in farming area which have a good opportunity to be developed in Indonesia. The development and role of dairy industries in Indonesia become one of the supporting factors of the development of dairy farm in Indonesia. Unfortunately, dairy industries' existence become a problem for dairy farmers. Harpini (2008) stated that 80% of milk coming from the farmer is absorbed by dairy industry, but it sometimes set the milk price that is less profitable to the farmers.

Dairy industries only accept milk from farmers that appropriate standard of quality. If the milk quality is above the standard of requirement, they will acquire above standard price, but if the milk quality is below the standard of requirement, the farmers will acquire below standard price. Dairy industries can also reject milk from farmers that has low quality (Martindah and Saptati, 2008). According to Zurriyati *et al.* (2011), fat and protein are milk nutrient that give impact on the milk selling price. Therefore, technological support in animal husbandry is needed, especially in post harvest handling process so, milk produced by the farmers have a good quality with high nutritional values to achieve optimal milk selling price.

The ultrafiltration membrane is a technology that is widely used for processing livestock products, one of them is milk. Ultrafiltration membrane technology can be used for milk nutrients concentrating process. Milk concentrating with membrane technology is conducted by separating the water and essential components using membrane (Kurniawan *et al.*, 2011). Separation using membrane technology can be done in low temperature so it can prevent the damage of milk nutrients which are sensitive to heat (Aspriyanto, 2002). Membrane technology have several advantages such as simple, which it does not need additional chemical substances and it has very minimum energy necessary (Kurniawan *et al.*, 2011).

The problems that often occur in filtration process using membranes are the fouling phenomenon. Fouling can decline of permeate flux because of the collection of material around or inside the membrane that made the membrane pores blocked or narrowed (Warsa, 2006). According to Susanto and Ulbricth (2009) one of the way that can be done to minimalize the risk of fouling is by mixing polymer membrane with additives in membrane manufacturing.

In this research, *polyethersulfone* (PES) was used as the polymer and *polyethylene glycol* (PEG) as the additive in ultrafiltration membrane manufacturing. PES is polymer that applicate widely in food manufacturing industry (Cao *et al.*, 2010). PES was chosen as polymer in the membrane manufacturing because it has a good durability toward chemical substance, has a good strength, tolerant toward temperature, and has a good stability (Qu *et al.*, 2010). PEG was used as the additive because based on the research done by Rosnelly (2012), the addition of PEG in the membrane manufacturing could increase the membrane flux, so membrane performance could be better. This study was investigated the effect of PES polymer concentration and time of filtration to the membrane performance (flux), water content, fat content, and protein content in milk.

II. Experimental

This research was conducted in Department of Chemical Engineering, Faculty of Engineering, Diponegoro University, Semarang. The materials used in this research consist of raw milk, PES from Solvay Advanced Materials, PEG 4.000 was supplied from Sigma Aldrich Company, N-methyl-2-pyrolidone (NMP) was purchased from Merck and aquades. Raw milk was obtained from morning milking in Faculty of Animal and Agricultural Sciences, Diponegoro University, Semarang.

The experimental design used in this research was split plot in time with five repetition. The main plot was filtration time consisting of T0 (before filtration), T1 (first hour of filtration), T2 (second hour of filtration), T3 (third hour of filtration), T4 (fourth hour of filtration) and T5 (fifth hour of filtration). The subplot was level of PES polymer concentration consisting of M1, M2 and M3. This research consisted of three steps, those were the membrane manufacturing, membrane application for milk filtration, and evaluation of milk nutrients.

PES Ultrafiltration Membrane Manufacturing

Membrane manufacturing was started by preparation of casting solution. The casting solution consisted of PES as polymer, PEG as additives, and NMP as solvent. The formulation of casting solution is shown in Table 1.

Table 1. Formulation of Casting Solution

Membran	PES Concentration	PEG Concentration
M1	13%	5%
M2	14%	5%
M3	15%	5%

The membrane casting was done using phase inversion method that was by casting the membrane on a glass plate using casting knive. Then, membrane immersed into coagulation bath containing aquadest for 1 hour, followed into different coagulation bath for 24 hours. Subsequently, membrane dried by aeration then continued with drying by oven with 106^{0}C temperature for 45 minutes.

The Application of PES Ultrafiltration Membrane for Milk Filtration

Milk filtration was done using filtration apparatus series as shown in Figure 1.

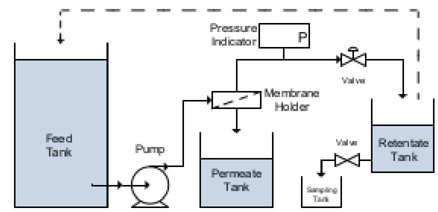

Figure 1. Unit Filtration Series

Milk was put into the feed tank. The membrane was cut with diameter of 4,2 cm and placed into membrane holder. Milk sampling was done before filtration (T0) and continued by taking sampling of milk from retentate tank every 1 hour (T1, T2, T3, T4 and T5). During the filtration process, membrane flux was tested to determine membrane performance. Flux testing was also done every 1 hour (started from T1, T2, T3, T4 and T5) by measuring permeate volume. This filtration process was performed for 5 hour. Membrane flux was counted using the equation:

$$J = \frac{V}{A.t} \qquad (1)$$

J = flux value (L/m2.hour)
V = permeate volume (L)
A = surface area (m2)
t = time (hour)

Evaluation of Milk Nutrients

Sample of milk before and after the filtration waere analyzed using lactoscan. The milk nutrients evaluated were water content, fat content, and protein.

III. Results and Discussion

The Effect of Polymer Concentration and Time of Filtration to the Ultrafiltration Membrane Performance

The membrane performance evaluated in this research was the flux. The PES ultrafiltration membrane flux value is shown in Table 2.

Table 2. Flux Value of Ultrafiltration Membrane in Different Polymer Concentration and Time Filtration

Filtration Time	Membrane			Mean
	M1	M2	M3	
--(Hour)--	-------------------Log L/m^2.hour----------------			
T1	1,61	1,51	1,46	1,53^a
T2	1,40	1,33	1,28	1,34^b
T3	1,33	1,19	1,18	1,23^c
T4	1,25	1,09	1,06	1,13^d
T5	1,15	1,00	0,91	1,02^e
Mean	1,35^p	1,22^q	1,18^{qr}	

The different superscript a, b, c, d and e in the same column shows a very significant difference (P<0,05).
Superscript p, q and r in the same row shows a very significant difference (P<0,05).

The result of analysis of variance showed there was no interaction effect (P>0,05) between the level of polymer concentration and time filtration. The level of polymer concentration treatment give significant effect (P<0,05) to the ultrafiltration membrane's flux value. In Figure 2, it can be seen that the higher the PES polymer concentration, the value of flux will get lower. This was because of the higher the concentration of polymer used in the membrane manufacturing, the smaller membrane pores formed, so the flux value will get smaller. In this research did not do Scanning Electron Microscope (SEM) analysis to determine membrane pore size, but based on the concentration of polymer, the M1 membrane was guessed to have the biggest pores size and M3 membrane was guessed to have the smallest pores size, whereas the M2 membrane was supposed to have pore size between pore size of M1 membrane and M3 membrane. This result is in line with Mulder (1996) who stated that the formation of pores in the membrane is affected by concentration of polymer in casting solution. The higher the polymer concentration, the denser the membrane's pore formed. In the study done by Sofiah *et al.* (2010) showed that the higher concentration of polymer, the smaller the membrane's flux.

The PES is a hydrophobic membrane polymer with low permeability, so it will be faster for fouling to happen. In the manufacture of mambrane from various concentration of PES and added by PEG as additive in the same concentration, the highest flux value is observed in membrane with the lowest PES concentration (Balamurali and Preetha, 2014). According to Wardani (2013), the addition of PEG is functioned to increase the hydrophilicity of the PES membrane. In a hydrophilic membrane, water will get into the membrane's pore faster than the hydrophobic membrane so, the flux rate will increase.

Flux value also can be influenced by component inside the feed. Based on the study done by Piluharto *et al.* (2013) showed that water water flux was higher than milk flux in flitration process used the same membrane. This is because of milk contained solid such as fat, protein, lactose and ash (Wibowo *et al.,* 2013). The more solid inside feed, the faster fouling happened so, membrane flux become decrease (Notodarmojo *et al.,* 2004).

The longer the filtration time, the lower flux value of PES ultrafiltration membrane. Based on the analysis of variance result, there was significantly different (P<0,05) in the time of filtration treatment to the value of membrane flux. In Figure 2, it can be seen that the longer the filtration time, the flux value is getting low. This was caused by the fouling at the membrane that increase as the filtration time gone by. It was in accordance with Zulfi *et al.* (2014) who stated that the longer the filtration time, the more particles stuck in the membrane surface which causes the decrease in flux of the membrane.

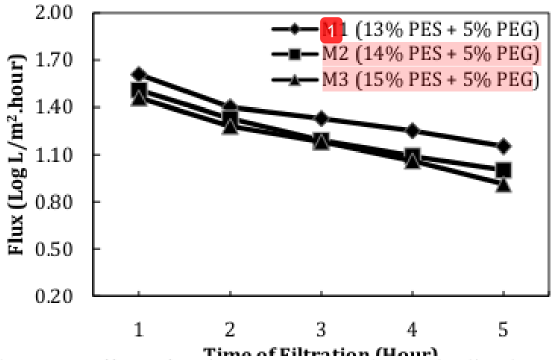

Figure 2. Effect of Polymer Concentration and Filtration Time to Flux Value

The Effect of PES Polymer Concentration and Time of Filtration to Milk Water Content. The result of water content analysis in milk filtrated using PES ultrafiltration membrane is shown in Table 3. Based on the analysis of variance, showed that there was no interaction effect (P>0,05) between the level of polymer concentration and time filtration to milk water content. The level of polymer concentration treatment gave significant effect (P<0,05) toward milk water content, while in the treatment of filtration time did not show significantly different (P>0,05).

Generally, in the filtration process used M1 and M3 membranes the water content were increased, while at M2 membrane the water content was decreased. The change of water content during the filtration process using M1, M2, and M3 membrane is shown in Table 4. In Table 4, it can be seen that M3 have a bigger increase in water content than M1, while M2 have a decrease in water content. If they are compared to the percentage of the milk water content in Table 3, the milk filtrated with M1 membrane have the highest water content. This was because of the milk that was being feed in the filtration process using M1 membrane has higher water content than milk that was being feed into M2 and M3 membrane, so the water content percentage in milk filtrated using M1 membrane had the highest value. This is in accordance to Kurniawan *et al.* (2011) who stated that the water content percentage produced from milk with low water content, in the same pressure will produce lower water content percentage.

The change in water content percentage happened through the filtration process could be impressed by membrane pores size, membrane properties, and also components inside the feed. PES membrane had a hydrophobic properties so, it had a low permeability. According to Radiman *et al.* (2002), hydrophobic membrane has a permeability that is not too good. The strength of hydrophobicity of membrane was influenced by the PES polymer concentration used in the membrane manufacturing. Based on the research done by Stefan *et al.* (2011), the higher the PES polymer concentration used in membrane manufacturing, the stronger hydrophobicity of membrane. The effect of PES concentration on Milk water content is tabulated in Table 3.

Table 3. Percentage of Milk Water Content Filtrated by PES Ultrafiltration Membrane with Different Polymer Concentration and Time Filtration

Filtration Time	Membrane			Mean
	M1	M2	M3	
--(Hour)--	-----------------------	(%)	-----------------------	
T0	90,37	89,39	89,66	89,81
T1	90,27	89,28	89,81	89,79
T2	90,35	89,41	89,70	89,82
T3	90,31	89,41	89,75	89,82
T4	90,36	89,46	89,86	89,89
T5	90,49	89,32	89,93	89,91
Mean	90,36[a]	89,38[b]	89,79[c]	

The different superscript a, b, c, and d in the same column shows a very significant difference (P<0,05).

The percentage of milk water content filtrated using M1 and M3 membrane were increasing. M1 membrane was an ultrafiltration membrane with the biggest pores and has the lowest hydrophobicity. In the filtration process using M1 membrane, more water volume came out from the membrane and it allowed some milk solids with small particles like protein and lactose still could escape through the membrane as permeates or entered and retained in the membrane's pores. This was in accordance to Zulfi *et al.* (2014) view that the bigger the membrane's pores, then more permeates amount could pass through the membrane. But, as the filtration time passed, more solids were accumulated in the membrane's surface so, the membrane's pores are getting smaller and blocked (Kartika *et al.*, 2009). This caused more water got into the retentate tank so, the water percentage of milk in the retentate tank became high as presented in Table 4

Table 4. The Change of Milk Water Content During Filtration

Filtration Time	Water Content Change (Increase / Decrease)		
	M1	M2	M3
--(Hour)--	-----------------------	(%)	-----------------------
T1	(0,11)	(0,13)	0,16
T2	0,10	0,15	(0,12)
T3	(0,05)	(0,01)	0,06
T4	0,05	0,06	0,12
T5	0,15	(0,16)	0,07
Mean	0,03	(0,02)	0,06

Numbers in parentheses indicate a decrease in water content.

The M3 membrane was the membrane with the smallest pores and has the biggest hydrophobicity. This caused the M3 membrane's permeability became low so, only a small amount of permeates volume could pass through the membrane. According to Aryanti *et al.* (2013), the hydrophobicity of membrane causes the water flux become low and tends to cause the fouling. Aprilia and Amin (2011) stated that the solute adsorption in the membrane surface that can plug in the membrane pores is a hydrophobic interaction and hydrophobic membrane was very susceptible toward fouling. This caused the water became more difficult to be separated so, the more water percentage got into the retentate tank. The effect of polymer concentration and time of filtration is presented in Figure 3.

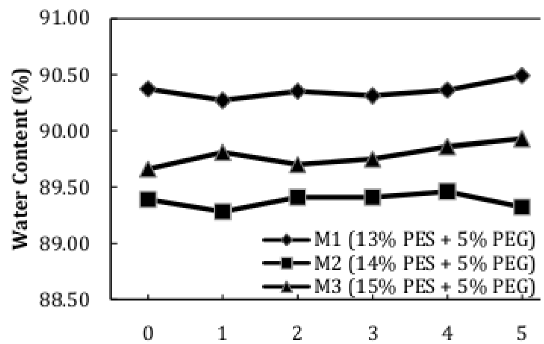

Figure 3. Effect of Polymer Concentration and Filtration Time to Milk Water Content

The water percentage of the milk filtrated using M2 membrane was decreased. M2 membrane had smaller pores than M1 membrane so, it could retain more milk solids than M1 membrane. According to Mulder (1996) membrane with smaller pore size can give a bigger repulsion compared to membrane with bigger pore size. The M2 membrane had lower hydrophobicity than M3 membrane. Membrane with low hydrophobicity can reduce the occurrence of adsorption solute or macromolecule (Ko *et al.*, 1993; Koehler *et al.*, 1997; Susanto *et al.*, 2012). This could reduce the fouling rate in M2 membrane so, the water in the milk could pass through the membrane and more milk solids could get into the retentate tank.

The Effect of PES Polymer and Time of Filtration toward Milk Fat Content

The analysis result at the filtrated milk using the PES ultrafiltration membrane is shown in Table 5.

Table 5. Percentage of Milk Fat Content Filtrated by PES Ultrafiltration Membrane with Different Polymer Concentration and Time Filtration

Filtration Time	Membrane			Mean
	M1	M2	M3	
--(Hour)--	-----------------------	(%)	-----------------------	
T0	2,84	3,03	2,55	2,81
T1	2,66	3,04	2,55	2,75
T2	2,56	2,91	2,55	2,67
T3	2,61	2,90	2,53	2,68
T4	2,65	2,83	2,43	2,64
T5	2,59	2,84	2,39	2,61
Mean	2,65	2,93	2,50	

Based on the analysis of variance, showed that there was no interaction effect (P>0,05) between the level of polymer concentration and time filtration to milk fat content. The polymer concentration and filtration time also did not give significant effect to milk fat (P>0,05). The fat content in the filtrated milk using the M1, M2, and M3 membrane tend to decrease, but that decrease was not showing any significant result. This was because of the most of fat in milk could be retained by the membrane, so that it could pass as retentate. Hariono *et al.* (2011) stated that milk fat has 0,92 – 15,75 µm size, while according to Mulder (1996), ultrafiltration membrane has 0,001 – 0,1 µm pore size. Figure 4 is shown the effect of polymer concentration on the milk fat content.

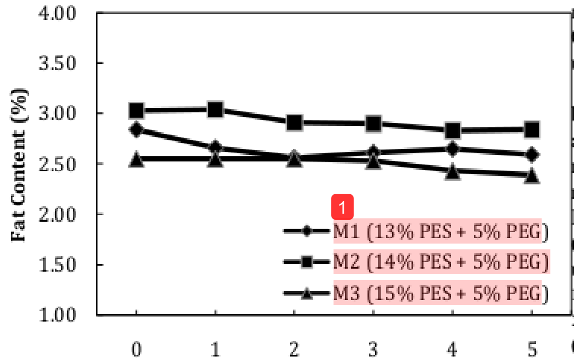

Figure 4: Effect of Polymer Concentration and Filtration Time to Milk Fat Content

The decreasing of milk fat during the filtration process was affected by the strong interaction between milk fat and membrane so, the fat adsorption was quickly formed in the membrane' surface. Xu *et al.* (2004) stated that fat is the fastest component in causing fouling in the membrane's surface. Richest *et al.* (1974) in Jian (1994) stated that in fat globule membrane there are phospholipoproteins that could adsorbed the membrane strongly because of the existence of amphoteric and amphiphilic particles that is strong enough to cause the irreversible fouling on the membrane. As the longer filtration time, the fat fouling in the membrane's surface was getting thicker so, there are less fat getting into the retentate tank. According to Rao (2000) in Chollagi (2009) fouling is a phenomenon that often happen to the ultrafiltration of dairy product. Fat content in dairy product can cause fouling on the hydrophobic membrane (Hausmann *et al.*, 2013).

The Effect of PES Polymer and Time of Filtration toward Milk Protein Content

The analysis result in the filtrated milk using PES ultrafiltration membrane is shown in Table 6.

Table 6: Percentage of Milk Protein Content Filtrated by PES Ultrafiltration Membrane with Different Polymer Concentration and Time Filtration

Filtration Time	Membrane			Mean
	M1	M2	M3	
--(Hour)--	------------------------(%)------------------------			
T0	2,75	2,77	2,85	2,79
T1	2,83	2,81	2,80	2,81
T2	2,82	2,81	2,83	2,82
T3	2,82	2,81	2,82	2,82
T4	2,83	2,82	2,82	2,82
T5	2,80	2,87	2,81	2,83
Mean	2,81	2,82	2,82	

The result of analysis of variance, showed that there was no interaction effect (P>0,05) between the level of polymer concentration and time filtration toward the milk protein content. It also showed there was no significantly different in the treatment of polymer concentration level and filtration time toward milk protein content (P>0,05). This was because of the protein contained in the milk filtrated using M1, M2, and M3 membrane could be retained by the membrane. According to Horne (2011) the milk protein has 0,05 – 0,6 µm size, while according to Mulder (1996), ultrafiltration membrane has 0,001 – 0,1 µm size.

The Table 6 shows that protein content of milk filtrated by M1, M2, and M3 membrane until the fifth hour are 2,59% at M1 membrane, 2,84% at M2 membrane and 2,39% at M3 membrane. The change of protein yield after filtration was not too big compared with the result of study done by Domagala and Kupiec (2003) and Moreno-Montoro *et al.* (2015). Domagala and Kupiec (2003) found that milk before ultrafiltration process containing of 3,25% protein and after filtration was obtained milk which is containing of 5,49%, 5,34% and 5,22% protein. Whereas, Moreno-Montoro *et al.* (2015) found that milk before ultrafiltration process containing of 4,10% protein and after filtration there were increasing of protein. Milk after ultrafiltration process containing of 5,73% protein. Kukučka dan Kukučka (2013) stated that there are many factors affecting filtration using membrane such as type of membrane, temperature of operation, feed, pressure and molecular weight cut off (MWCO). Effect of polymer concentration on the milk protein content is presented in Figure 5.

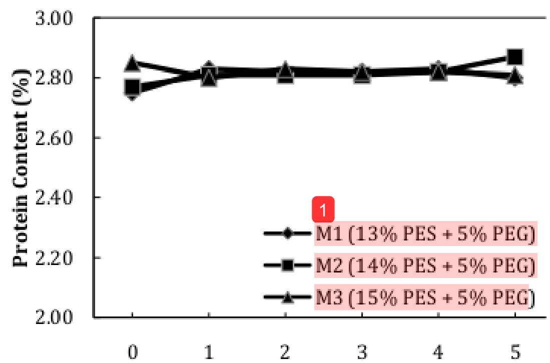

Figure 5: Effect of Polymer Concentration and Filtration Time to Milk Protein Content

The percentage of protein content could be influenced by the increase in milk water content. The higher the water content in the milk, the lower the solids composition in the milk (Kurniawan *et al.*, 2011). Besides, the hydrophobicity of membrane can affect the percentage of milk protein content. The hydrophobicity in membrane caused the adsorption of the protein in the membrane so, more protein accumulated and coated the membrane's surface. This caused the protein that pass to the retentate tank decreasing. In its structure, protein has both hydrophobic and hydrophilic clusters (Mc Clements, 1999; Pahlevi *et al.*, 2008). The protein adsorption mechanism to the membrane's surface happen because of the pulling force from the hydrophobic protein part to the hydrophobic membrane's polymer surface (Piluharto *et al.*, 2013). It was strengthened by Liu *et al.* (2012) who stated that hidrophobic interaction and electrostatic between protein and PES ultrafiltration membrane can cause adsorption of β-lactoglobulin on membrane surface.

IV. Conclusion

Based on the results of study, it could be concluded that there was no interaction effect between the level of polymer concentration and time filtration toward the membrane flux and milk nutrient (water, fat and protein content). The difference of PES polymer concentration significantly influenced membrane flux and the milk water content, while the filtration time only give significant effect to membrane flux. The difference of PES polymer concentration and the time filtration did not give a significant effect toward fat and protein in the milk. The best PES ultrafiltration membrane for milk filtration was the M2 membrane that containing 15% PES polymer and 5% PEG with 5 hours filtration time because it had potential to reduce milk water content and increase milk protein content.

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