Analysis of fouling and juice quality in crossflow ultrafiltration of watermelon juice

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Abstract

Raw watermelon juice was clarified in a laboratory scale flat plate ultrafiltration system incorporating polyethersulphone membranes with a molecular weight cut-off of 50 kDa. The experiments have been carried out over a wide range of trans-membrane pressures (1-3 bar). The effect of ultrafiltration with molecular weight cut-off (MWCO) of 50 kDa PES membrane on permeate properties is reported. The effects of trans-membrane pressure (TMP) on permeate flux and resistances were studied. The flux decay was analyzed through the resistance in series model which showed increase of both reversible and irreversible resistance with trans-membrane pressure, The TSS content of the permeate was found to be almost the same with feed while the ascorbic acid content in the permeate was on the lower side as compared to in feed.

Keywords: watermelon juice; ultrafiltration; fouling; permeate flux; resistance in series model.

Practical Applications: As watermelon is a seasonal fruit, to make it available all through the year, the juice has to be processed in order to store it for longer periods. Membrane technology provides the best option to treat the juice in an athermal manner to retain its nutritional and sensorial characteristics after processing. Ultrafiltration has proven to be a great tool for clarifying fruit juices in recent times. Watermelon has a significant amount of vitamin c and also contains lycopene, a carotenoid with potential cancer preventive properties. Ultrafiltration can be useful in retaining these important components in the clarified juice and reduce the amount of water and suspended solids, thereby increasing its shelf life.

1 Introduction

During the last two decades, researchers have broadly studied fruit and vegetable juice clarification using ultrafiltration. Ultrafiltration, over the years has been successful in replacing the use traditional thermal techniques for the clarification of fruit juices. UF is capable of retaining macromolecules such as proteins, tannins, and polyphenols in the retentate while micro solutes, for example sugars, vitamins, organic acids, aroma compounds, pigments **and salts** permeate through the membrane along with water. Since it is a low temperature operation, chances of microbial activity damaging the permeate is very minimal compared to thermal evaporation techniques. The retentate or concentrate part produced by UF is made up of fibers and suspended solids and the permeate is mainly water plus essential micro molecules which is free of nonessential microorganisms. It has many advantages over traditional thermal techniques, some of them are low operating temperature, low energy requirement, thus minimizing associated costs, no use of fining agents and additives, better product quality, low amount of waste generated, single step continuous process, less process completion time, simpler process design, less manpower requirement, increased juice yield, simple cleaning methods and maintenance of the equipment (Girard et al., 2000). Clarification of different fruit juices using UF results in color improvement in the juice and it also increases the clarity of the juice by eliminating pectic

substances and colloidal particles. The clarified juice has a lower viscosity due to the removal of macromolecules and suspended solids. The soluble solids which are mainly sugar and vitamins are recovered in permeate. The pH and density are generally not affected by juice filtration using UF (De Bruijn et al., 2003; Laorko et al., 2010; Matta et al., 2004). The most widely used membrane modules for the clarification/concentration of fruit juices are tubular (Vladisavljevic et al., 2003), hollow fibre (Tasselli et al., 2007), plate-and-frame (Mirsaeedghazi et al., 2007) and flat plate (Bhattacharjee et al., 2017) membrane modules.

Watermelon is a readily available inexpensive fruit in summer season in India. It is mainly served as slices or chunks and juice. Although it is a summer fruit, but is becoming highly consumed like everyday fruits such as apples, bananas and oranges. It consists of 90% water, with little quantity of protein, fat, minerals and vitamins. India is a warm country and temperatures rise above 40 °C in most parts during the summer season and watermelon is used as a valuable source of water in these regions. Due to its tropical nature, its production is high during the summer and it is available from March to September in India depending upon the location. Watermelon production occupies 6-7% of overall fruit production (Reddy et al., 2008). Sensorial and nutritional properties make this juice hugely popular among masses (Edwards et al., 2003). Vitamin C and

phenolic content of watermelon juice is a bit low compared with other fruits (Gil et al., 2006). Research has shown that Vitamin C helps in fighting cancer. It also helps in improving plasma cholesterol concentration levels, and improves balance to collagen associated disorders in the body (Block, 1991; Kurowska et al., 2000). Watermelon juice contains significant proportions of lycopene, a bright red carotenoid compound imparting red color to it (Perkins-Veazie et al., 2001). Nutritional aspect of lycopene has been researched a lot in recent times and it has been proven that it can lessen the risk of various types of cancers and also able to prevent cardiovascular disease. Lycopene has the capability of blocking the proliferation of four types of malignant cells (Salman et al., 2007). Review of Giovannucci (1999) **revealed** that lycopene effectively decreases the possibility of lung, stomach, and prostate cancers while some research put forwards information that lycopene may have the ability to minimize the risks of cervix, breast, oral cavity, pancreas, and esophageal cancer.

2 Materials and methods

2.1 Juice preparation

Fresh watermelons were bought from the nearby local market of Indian Institute of Technology (Indian School of Mines), Dhanbad. Before processing, they were thoroughly washed with water, and then chopped and deseeded manually. The fruits were sliced in various pieces according to the juice requirements and then extraction of seeds and pulp was done. Then the juice was squeezed using a juicer and after that it was filtered by vacuum filtration for further removal of pulp.

2.2 UF experimental setup

The Ultrafiltration tests were accomplished in a flat plate UF unit incorporating a flat sheet membrane running in crossflow mechanism with pressure control gauges. The organic (polyethersulphone) membrane has an MWCO of 50 kDa and filtration area of 50 cm² (Sartorius, Kolkata). Freshly prepared juice was charged into the module using a peristaltic pump. The ultrafiltration was conducted at pressures of 1, 2, 3 bar respectively with 50 kDa. Permeate volume with time was noted at regular intervals for flux determination, and after the end of runs at different parameters, some portions of permeate and retentate were assembled for the measurement of physicochemical properties. The juice ultrafiltration was performed in line with batch concentration technique. According to batch concentration method, retentate was recycled to the feed tank and permeate is collected separately at different process parameters. The UF operations were performed at room temperature of 30 ± 1 °C. The characteristics of the flat plate UF module is listed in Table 1.

2.3 Membrane cleaning

The pure water flux(J) was measured by adding distilled water into the membrane module and by measuring the volume of permeate ($V_{permeate}$) collected in a certain time t through the membrane surface area A according to the Equation 1:

$$J = \frac{V_{permeate}}{t \times A}$$ (1)

Table 1. Characteristics of UF Membrane Module.

Parameters	Type
Configuration	Flat Plate
Polymer	Polyethersulphone
MWCO	50 kDa
Membrane Area	50 cm²
pH operating range	2-13
Operating Pressure	0.5-3 bar

where A is the effective membrane area and t is the permeation time. Before each experiment the membrane module was compacted at the highest operating pressure of 3.5 bar with distilled water at room temperature. For hydraulic permeability measurement, the pure water flux values were plotted against the applied transmembrane pressure (TMP) values. The slope of the generated straight line gave the hydraulic permeability of the UF membrane. This permeability value of the fresh membrane is referred to as L_p^0. The hydraulic permeability of the membrane after the UF of watermelon juice is denoted by L_p^1.

The cleaning procedure for the membrane module consists of two steps. At first, distilled water was recirculated for around 30 min at 3 bar through the membrane module so as to minimize the affect of reversible cake layer resistance. Again the hydraulic permeability was measured and is denoted as L_p^2. Subsequently, the module was submitted to a cleaning solution of 250 ml of 0.5 mM NaOCl (sodium hypochlorite) in 0.5 M NaOH (sodium hydroxide) for 30 - 40 minutes. After that the module was given a final rinsing with deionised water for 5-10 minutes and finally the module is fiiled with deionised water and 10% ethanol solution and stored in a refrigerator at approximately 4 °C. The extent of membrane fouling, which is linked to the percentage drop in the pure water flux after the filtration, was determined by the difference of pure water flux before the UF treatment of juice and after the cleaning process.

2.4 Watermelon juice characterization

The fresh juice, permeate and retentate samples from the ultrafiltration (batch concentration mode) of watermelon juice were collected and stored in a refrigerated state before performing analytical evaluations. The samples were examined to determine density, viscosity, acidity, pH, total soluble solid, color, clarity, ascorbic acid and lycopene.

Color measurements were done by determining the value of absorbance at 420 nm and clarity by transmittance at 625 nm with the help of an UV–Vis Spectrophotometer (Thermofisher Scientific). A digital refractometer (Mettler Toledo) was used for measuring total soluble solids (TSS). TSS measurements were performed at the standard temperature of 20 °C. pH was measured by pH meter **(Mettler Toledo)**. For titratable acidity measurement, titration of juice sample was performed with 0.1 N NaOH to pH 8.2 and the required volume (ml) was converted into malic acid equivalent (Ranganna, 1986) and was expressed as weight percent malic acid. Viscosity was measured by using a glass Ostwald capillary viscometer and the density was calculated with the help of a pycnometer. 2, 6- dichlorophenolindophenol

(DCPIP) titration was done for ascorbic acid determination (Association of Official Analytical Chemists, 1995).

2.5 Lycopene content assay

For lycopene content determination, methods of Sadler et al. (1990) and modification of Perkins-Veazie et al., 2001 were used. Extraction was done using a mixture of hexane/acetone/ethanol at a proportion of 2:2:1 (v:v:v). After extraction, the absorbance of the hexanic phase was measured at 503 nm. This wavelength provides minimal interference from other watermelon carotenoids (Holden et al., 1999; Zechmeister et al., 1943). The lycopene content was estimated by the following equation using a molar extinction coefficient equal to 17.2×10^4 L/mol/cm. The results were expressed as µg lycopene per g sample according to Equation 2:

$$Lycopene\ (\mu g / g) = \frac{\Delta_{503} \times MW \times V \times 1000}{\varepsilon \times b \times W} \tag{2}$$

Where

A_{503} = Hexanic phase absorbance at 503 nm;

ε = the molar extinction coefficient (L/mol/cm);

b = length of optical path (cm);

MW = molecular weight of lycopene = 536.9 g/mol;

W = the sample weight (g).

3 Results and discussion

3.1 Permeate flux analysis

Figure 1 shows the variations of the juice permeate flux with time in the range 1 - 3 bar. From the figure, rapid declination of permeate flux can be seen at the initial period followed by gradual reduction after that period. The flux trajectory comprises of two distinct zones. Rapid decrease of permeate flux in the first zone is associated with the deposition and generation of a reversible polarized layer established by macro solutes present in the juice (Rai et al., 2007). These higher molecular weight compounds are cellulose, hemicelluloses, polysaccharides, protein **and colloidal materials**. These solutes don't permeate and deposit on the surface with time and creates a secondary membrane like layer as ultrafiltration goes on and thereby adding reversible membrane resistance to the total membrane resistance. After that initial period of half an hour the polarized layer settles and dictates a gradual decline in flux attaining a steady state value.

In order to find out the effect of pressure on permeate flux, UF experiments were performed in total recycle mode. In Figure 2 the steady state permeate flux values are plotted against the applied TMP at a fixed value of flow rate (300 ml/min) and at a fixed temperature of 30 °C. The shear forces generated by low pressure are well capable of preventing solute deposition over the surface and therefore in this low pressure region, the flux is almost proportional to the applied TMP. This region is called the pressure controlled region. But, after a certain point, an increase in pressure seems inconsequential as flux tend to approach a limiting value and stops increasing with
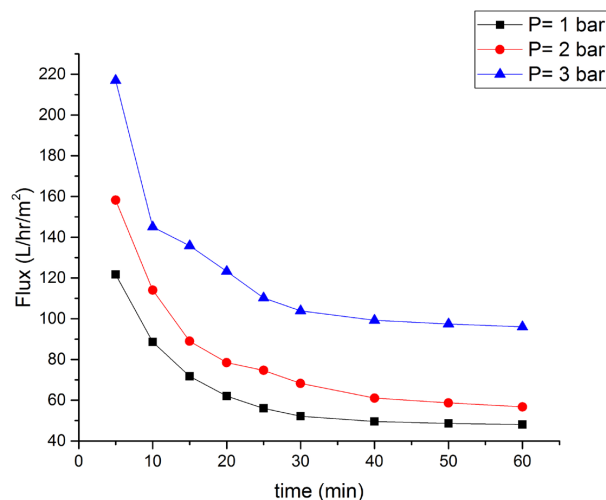

Figure 1. Time course of permeate flux at different pressure values.
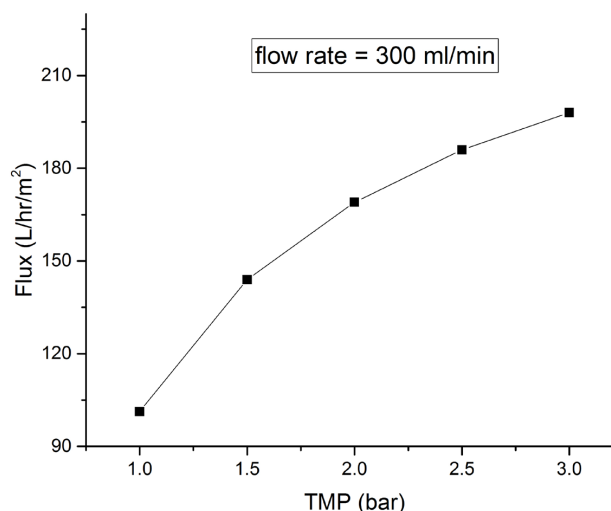

Figure 2. Effect of applied TMP on permeate flux.

further increase in TMP. This region is called the mass transfer controlled region as concentration boundary layer starts dictating the flux pattern instead of applied pressure. Rejected macro solutes generate this concentration profile across the membrane surface. Further increase in pressure helps in consolidation of this concentration layer leading to the formation of a secondary membrane layer, thus adding an additional resistance to the permeate flux (Cassano et al., 2007). The pressure at which the permeate flux was maximum is considered the optimum TMP for the clarification process which is 3 bar in this case.

The crossflow feed velocity is directly related to mixing and shear stress inside the membrane module as an increase in flow rate reduces concentration polarization and enhances mass transfer and as a consequence helps in removing deposited particles over the surface. This directly causes an increase in permeate flux. The influence of feed flow rate on permeates flux at a trans-membrane pressure of 3 bar and temperature of 30 °C is shown in Figure 3. The flux increases with increasing velocity due to the increased shear at the surface of the membrane

3.2 Fouling resistance analysis

Resistance in Series Model-

The intrinsic membrane resistance is determined with the help of Equation 3:

$$R_m = \frac{\Delta P}{\mu_w \times J_w} \tag{3}$$

In the above equation, R_m is the intrinsic membrane resistance, ΔP is the TMP (bar), μ_w is water viscosity (cP), and J_w is denoting the pure water flux of the membrane before the beginning of the experiment (L/hr/m^2).

The resistance generated by membrane fouling can be evaluated with Equation 4:

$$R_F = \frac{\Delta P}{\mu_p \times J_p} - R_m \tag{4}$$

where μ_p (cP) is the watermelon permeate viscosity and Jp (L/hr/m^2) is the permeate flux. R_F is the membrane fouling resistance.

It comprises of reversible fouling (R_{frev}) which is offered by the concentration layer formed over the surface and irreversible fouling resistance (R_{firr}) due to pore blocking phenomenon. Reversible and irreversible fouling resistances are measured as shown in Equations 5 and 6:

$$R_{firr} = \frac{1}{\mu_w \times L_p^2} - R_m \tag{5}$$

$$R_{frev} = \frac{1}{\mu_w \times L_p^3} - R_m - R_{firr} \tag{6}$$

where L_p^2 and L_p^3 are hydraulic permeabilities (water flux divided by transmembrane pressure) after washing with water and chemicals and after washing with water, respectively.

The watermelon juice was treated at various pressures to see the effect of pressure on resistances. Figure 4 shows the influence of ΔP on total, reversible and irreversible layer resistance during the ultrafiltration process. From the figure it can be seen that the reversible and total resistance increases significantly as the trans-membrane pressure increases. This happens mainly because of the convection of solutes from bulk towards the membrane surface. Due to this more solutes are deposited on the membrane surface, increasing the thickness of the layer, thereby increasing concentration polarization which in turn enhances the reversible resistance. At higher pressures the amount of solute such as sugars and acids passing through the membrane increases resulting in the increase of fouling resistances.

The effect of individual membrane resistances and the ratio of reversible and irreversible resistance to the total resistance at different transmembrane pressures is shown in Figure 5. At 1 bar the difference in contribution of the individual resistances to the total resistance was very less. As the pressure increased that difference rose significantly, R_{rev} having the main contribution towards the total resistance. It also increased linearly while the other two (R_m & R_{irr}) decreased slowly. At 3 bar R_{rev} contributed more than half (54%) to the total resistance while the R_{irrev} contribution was modest (27.5%). The contribution of intrinsic
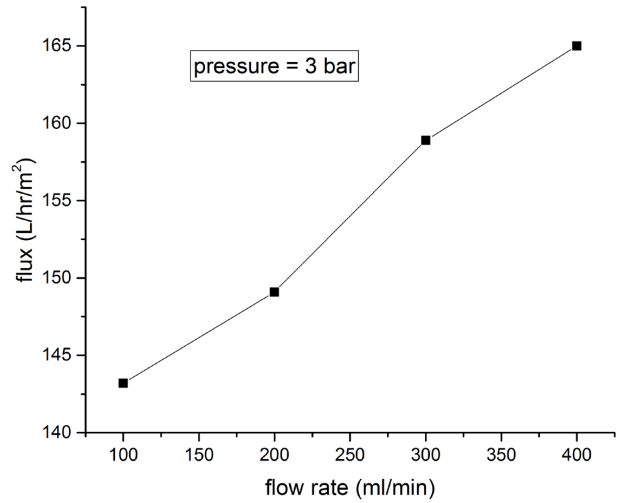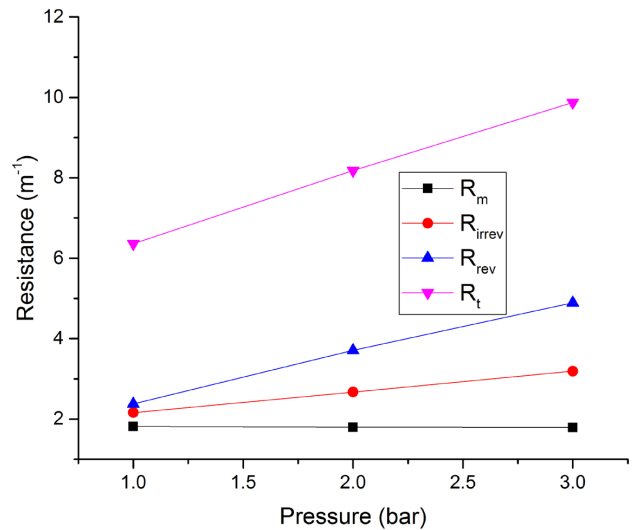

Figure 3. Effect of flow rate on permeate flux.

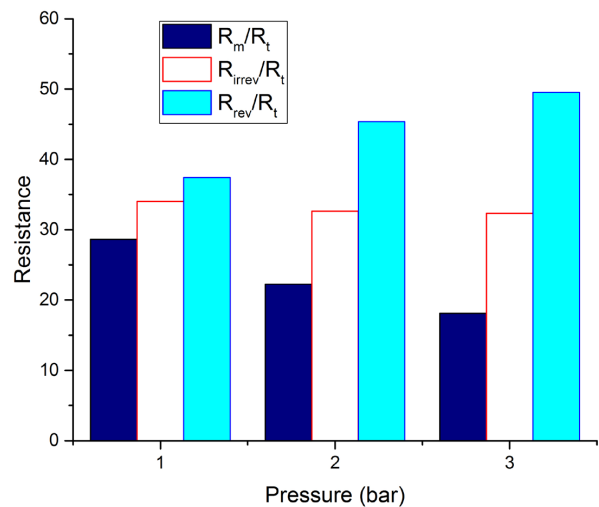**Figure 4**. The effect of ΔP on membrane resistances.

Figure 5. Contribution of resistances at different pressures.

membrane resistance decreased from 29.6% to 18% as the pressure increased from 1 to 3 bar.

3.3 Cleaning efficiency

The hydraulic permeability of the PES membrane was calculated using the pure water flux and trans-membrane pressure data and was found to be 216 L/m^2 h bar. Membrane resistance was found to be 1.8×10^{12} m^{-1}. The hydraulic permeability was recovered up to 86% by cleaning after the watermelon juice clarification process. Figure 6 shows the pure water permeate flux before and after the Ultrafiltration and cleaning process. The hydraulic permeabilities of the PES membrane before and after cleaning procedure is measured and it can be seen that the permeability dropped by 47% after the juice clarification. After washing with water it was mildly recovered. But after cleaning with alkali and hypo solution the permeability was regained by almost 86%. Thus we can say that the cleaning procedure using **NaOH and NaOCl** was quite successful in removing the reversible fouling from the surface of the membrane.

4 Analytical evaluations

The UF process was quite successful in removing suspended solids from freshly produced watermelon juice. Refractometer readings of total soluble solids (TSS) content were on the higher
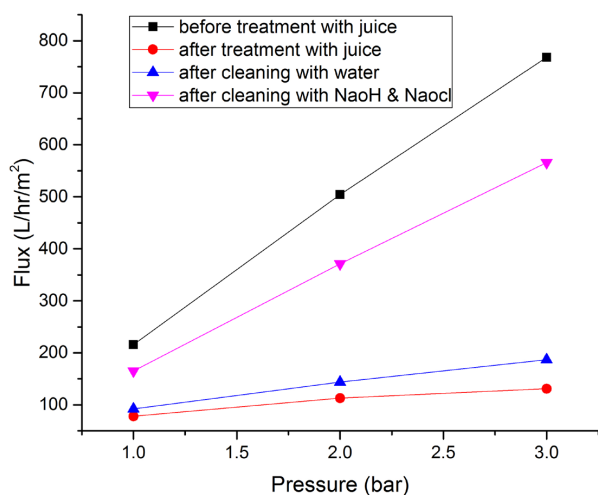

Figure 6. Pure water flux of membrane before and after UF and cleaning treatments.

Table 2. Physicochemical Properties of Feed and permeate.

Parameters	Feed	Permeate
Density (kg/l)	1.08	1.03
Viscosity (cp)	0.97	0.91
pH	5.13	5.01
TSS (^{0}Brix)	7.1	6.9
Acidity (wt% malic acid)	0.078	0.07
Color (%A$_{420}$)	3.16	0.245
Clarity (%T$_{660}$)	2.11	83.17
Ascorbic Acid (mg/l)	59.83	53.12
Lycopene (mg/l)	35.51	12.15

side for retentate. This aspect can be described by the fact that there is a high amount of suspended solids which is rejected by the membrane along with the pulp which ultimately interfere with the refractive index measurements (Cassano et al., 2007), although there is only a slight reduction of TSS in permeate which is around 3%. The acid is also mostly recovered in permeate. There is a significant improvement of clarity in permeate as it mostly contains water. The permeate viscosity reduced by 15%. In the clarified juice, ascorbic acid reduction was observed around 12% with respect to the fresh juice which can be linked with the oxidation of this component caused by continual recycling of the juice around the Ultrafiltration loop (Cassano et al., 2007). Lycopene was mostly recovered in the retentate stream which can be processed further to purify it. The clarification process didn't have much effect on the pH of the juice.

Table 2 reports the physicochemical properties of the feed and permeate of the UF process.

5 Conclusion

Steady-state, crossflow UF was used for clarification of watermelon juice. The ultrafiltration was performed in a pressure range of 1-3 bar. The results of this study showed that ultrafiltration can clarify watermelon juice without causing significant changes in its important physicochemical properties **with minor reductions of 2.8% and 11.2% w.r.t. soluble solid and ascorbic acid content** respectively. Irreversible fouling presented minimal contribution to the total resistance (27.5%) compared to the contribution of the reversible fouling, which was more significant (54.1%). Reversible resistance increased significantly when the transmembrane pressure was increased. Alkali and hypo solutions used for performing chemical cleaning proved to be quite successful in removing the polarized reversible layer and restored about 86% of the initial hydraulic permeability of the membrane. Therefore, a combination of suspended particles and adsorbed macro solutes were mainly responsible for the extent of fouling in the UF process.

The physicochemical and nutritional properties of the clarified juice are very much comparable with those of fresh juice. Ultrafiltration of watermelon juice allowed an acceptable level of clarification in terms of sugar and ascorbic acid and also greatly reduced the suspended solids and the turbidity of the fresh juice. A 12% reduction of the total ascorbic acid was measured in the permeate stream with respect to the fresh juice and a high percentage of soluble solids were retained in permeate. The retentate portion can be utilized to extract the valuable antioxidant lycopene.

Abbreviations

MWCO Molecular weight cut-off

UF Ultrafiltration

TMP Transmembrane Pressure

TSS Total Soluble Solids

PES Polyethersulphone

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