Advancement In Forward Osmosis (FO) Membrane For Concentration Of Liquid Foods

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Abstract. In food processing, concentration of liquid food is one of the important steps required for several purposes. Concentration of liquid food while preserving sensorial and nutritional components is quite challenging, especially for thermal-based concentrating processes. This is due to the significant loss of those components which are heat sensitive. Therefore, considerable efforts have been devoted to develop new concentrating processes which can solve this problem. Among the developed processes, forward osmosis (FO) has been considered as an interesting alternative since it can be operated at low operating pressure and temperature and obtain a concentrated solution with high solid contents. However, there are several challenges in FO operation e.g. fouling phenomena, concentration polarization, and reverse diffusion of solution from draw solution. To address these issues, several developments have been made to prepare membrane which has high hydrophilicity, low fouling tendency, reduced concentration polarization, and low solute diffusion. The desired membrane has been obtained, for example, by modifying selective and support layers of the membrane. This paper reviews advances in FO membrane, including membrane preparation and modification. Principle and important parameters of FO in concentrating liquid foods are overviewed. In addition, challenges and strategies in FO membrane preparation are discussed.

Keywords: food processing; liquid concentration; membrane; osmosis;

1. Introduction

Liquid food concentration is one of the important steps in food processing. The aim is to maintain product stability by reducing water content. The liquid food is generally concentrated by thermal-based processes. However, it is hard to keep the nutritional value of the food in such processes. Most nutritional components are heat sensitive thus exposing these components to elevated temperature results in degradation of bioactive compounds [1,2]. On the other hand, there is an increasing demand on healthy food or functional food due to an improved paradigm aiming for healthy lifestyle. This trend has attracted researcher from both academia and industries to find the alternative process for liquid food concentration which
could preserve the nutritional value [3].

Forward osmosis (FO) is considered as an attractive process which can address the drawbacks of the conventional concentration process. As shown in Figure 1, there is a significant increase of publications related to liquid concentration with FO in the last decade. The increasing attention on FO studies was driven by the lower energy consumption and enhanced performance [4]. FO is a membrane-based process which utilizes osmotic pressure as the driving force and semi-permeable membrane as a selective barrier. Osmotic pressure originated from a different solute concentration drives the permeation of pure water from a low salinity solution (feed solution, FS) to high salinity solution (draw solution, DS). The osmotic pressure difference is created by employing a DS which has a higher solute concentration than the FS. FO offers several interesting features including a low operating pressure, ability to treat a high solid content feed, and preserve nutritional compounds due to mild operating condition [5–7]. FO exhibits several advantages compared to pressure-driven membrane processes (reverse osmosis, nanofiltration, etc.). The advantages of FO include no or low hydraulic pressure, high water recovery, lower fouling tendency, and higher energy efficiency [8]. Therefore, FO has gained increasing attention from researchers to explore those advantages indicated by the increasing reported studies in this field (Figure 1).

**Figure 1.** Number of publications related to forward osmosis indexed by Scopus and Web of Science, WoS (query: TITLE(terms); terms: forward osmosis, forward osmosis concentration)

Despite those advantages, FO still has some shortcomings that need to be solved. The first one is the internal concentration polarization (ICP) [9–13]. ICP reduces the osmotic pressure difference between the FS and DS which results in a decrease of water permeation rate or flux. ICP occurs in membrane support as the effect of unstirred boundary layer formed at the interface between the active and support layer. Reverse salt diffusion is another shortcoming of FO operation which also reduces the effectiveness of the process [14–16]. This phenomenon occurs due to imperfect membrane selectivity and the high salt concentration of DS. Reverse salt diffusion may reduce water flux due to DS dilution. Diffusion of salt from DS to FS can contaminate the product. Liquid foods contain various compounds that may have potentials to fouling occurrence. Fouling is the major drawback of membrane operation [17,18] which also occurs in FO operation [19,20]. Fouling reduces membrane productivity and increases membrane operating and maintenance costs. Those shortcomings are mainly related to the membrane characteristics. Therefore, there are considerable efforts to develop
membranes which could reduce those drawbacks.

Liquid food concentration with FO has been reviewed in several papers [5–7]. Therefore, this paper will be a complement and an update of the previous published papers. Our main focus in this review is on some advancements in FO membrane and FO process in liquid food concentration including performances and challenges.

2. FO for Concentrating Liquid Foods

Table 1 summarizes several reported studies on liquid food concentration using FO process. DS is one of FO important parameters which determines the effectiveness of FO process. DS should have high osmotic pressure, non-toxic, and easy to regeneration [21]. Petrotos et al. [22] employed FO for concentrating tomato juice and investigated several parameters affecting the FO performance. The performance was analyzed by observing water flux. They compared six DSs, including NaCl, CaCl$_2$, Ca(NO$_3$)$_2$, sucrose, glucose, and polyethylene glycol 400. Among the solutions, NaCl was found to be the best option due to its low viscosity. In general, the water flux was increased by solute concentration of DS since higher concentration led to a greater osmotic pressure. Results of their study also suggested using a thinner membrane because it resulted in a higher flux. Moreover, the water flux tended to increase exponentially with membrane thickness. Garcia-Castello and McCutcheon [23] compared two different DS concentration, i.e. 2 M and 4 M, for concentrating orange peel press liquor in FO. They found that the liquor could be concentrated up to 3.7 concentration factor when 4 M NaCl was used.

In FO, the membrane can be operated at two modes according to membrane orientation [24]. The first mode is called AL-FS mode in which the membrane active layer facing the feed solution (FS). Meanwhile, the second mode uses a membrane with the active layer facing the draw solution (DS) which is also known as AL-DS mode. Nayak et al. [25] evaluated effects of membrane orientation in FO and molecular weight compounds on FO performance, in term of water flux. According to their results, AL-FS could result in a higher flux rather than AL-DS mode. It was due to a significant external concentration polarization occurred in AL-DS mode. In another study, Petrotos et al. [26] tried to determine the effect of pre-treatment step on FO performance during tomato juice concentration. They proposed a pre-treatment step for clarifying the juice prior to FO. The result showed that pre-treatment step could effectively improve water flux above 100% compared to those unclarified juices. The highest flux was obtained when 25 kDa ultrafiltration membrane was used in the pre-treatment step. A pilot scale study reported by Dova et al. [27] showed membrane characteristics, solute concentrations of feed solution (FS) and DS, and flow rates of FS and DS affected FO performance significantly. Garcia-Castello and McCutcheon [23] compared DS with two different concentrations, i.e. 2 M and 4 M, for concentrating orange peel press liquor in FO. The 4 M NaCl DS could achieve concentration factor up to 3.7.
### Table 1. Performances of FO in liquid food concentrations

| Liquid foods            | Membrane                          | Draw solution                  | Operating conditions       | Results                          | Ref. |
|------------------------|-----------------------------------|--------------------------------|----------------------------|----------------------------------|------|
| Tomato juice           | Polyamide (AFC99, TFC)            | 22.2% wt. NaCl                 | Initial TSS = 4.3 °Brix;    | Flux = 3.1 kg m⁻² h⁻¹;          | [22] |
| Tomato juice           | Polyamide (AFC99, TFC)            | 23% wt. NaCl                   | Initial TSS = 4.3 °Brix;    | Flux = 7.28 kg m⁻² h⁻¹;         | [26] |
| Red Radish Juice       | Osmotek Inc., Corvallis, OR       | 60 °Brix fructose corn syrup   | Initial TSS = 1.2 °Brix;    | Final TSS = 5.1 °Brix (single process), 8.2 °Brix (double process). | [28] |
| Pineapple juice        | Cellulose acetate (Osmotek, Inc.) | Mixed 40% sucrose – 12% NaCl  | Initial TSS = 12.4 °Brix;   | Final TSS = 60°Brix;            | [29] |
| Glucose solution       | Polyamide membrane (DS-3-SG); TFC | 23% NaCl                       | Initial glucose conc. = 0.17 molality; | Flux = 1.7 kg m⁻² h⁻¹;         | [27] |
| Sucrose solution       | Cellulosic membrane; Albany, OR;  | 4 M NaCl                       | Initial TSS = 10 °Brix;     | Flux = 5.84 L m⁻² h⁻¹;          | [30] |
| Anthocyanin extract    | Cellulose acetate (Osmotek, Inc.) | 6 M NaCl                       | Initial TSS = 2 °Brix; Anthocyanin conc. = 49.6 mg/L; | Final TSS = 52 °Brix; Anthocyanin conc. = 2.7 g/L; | [31] |
| Beetroot juice         | Cellulose acetate (Osmotek, Inc.) | 6 M NaCl                       | Initial TSS = 2.3 °Brix; Betalains initial conc. = 50.92 mg/L; | Final TSS = 52 °Brix; Betalains final conc. = 2.91 g/L; Initial flux = 11-12 L m⁻² h⁻¹; | [25] |
| Grape juice            | Cellulose acetate (Osmotek, Inc.) | 6 M NaCl                       | Initial TSS = 8 °Brix; Anthocyanin content = 50.9 mg/L; | Final TSS = 54.6 °Brix; Final Anthocyanin content = 2.9 g/L; Initial flux = ~8 L m⁻² h⁻¹; | [25] |
| Tuna cooking juice     | Cellulosic membrane; Albany, OR;  | 2 M NaCl                       | Initial protein conc. = 5.5 % (w/v); | Final protein conc. = 9 % (w/v); Flux = 2.54 L m⁻² h⁻¹; | [32] |
| Jaboticaba (Myrciaria jambctica) juice | Cellulosic membrane; Albany, OR;  | 6 M NaCl                       | Initial TSS = 12 °Brix; | Flux = 3.2-3.59 L m⁻² h⁻¹; | [33] |
| Sweetlime juice        | Cellulose acetate (Osmotek, Inc.) | 6 M NaCl                       | Initial TSS = 11 °Brix;     | Initial TSS = 50 °Brix; Initial flux = 9-10 L m⁻² h⁻¹; | [34] |
| Raspberries juice      | Osmotek’s direct-osmotic concentration (DOC) units | 69.7 °Brix fructose corn syrup | Initial TSS = 10 °Brix; T = 26°C; t = 5.8 h; | Final TSS = 44.8 °Brix; Flux constant = 1.4 L m⁻² h⁻¹ Δ°Brix⁻¹; | [35] |

TFC – thin film composite; TSS – total soluble solids;

Concentrating bioactive-containing liquid while preserving the active function is highly needed in various industries. This ability has been performed by using FO in several studies. For example, Yang et al. [36] used FO for concentrating pharmaceutical products by employing a home-made dual-layer membrane. Lysozome product was effectively enriched by FO with a high purity and without properties change. Rodriguez-Saona et al. [28] investigated the performance of FO for the concentration of Red radish extract by employing corn syrup solution (60 °Brix) as the DS. The FO process could increase the total soluble...
solids (TSS) of Red radish extract from 1.2 °Brix to 5.1 °Brix in a single concentration process and to 8.2 °Brix in a twice concentration process. Furthermore, they also demonstrated that the concentration of TSS could be increased by combining FO with the conventional evaporator. In addition, the concentrated juice also showed an increase in Anthocyanin concentration. The final Anthocyanin concentration increased to 0.899 mg/mL from 0.258 mg/mL initial concentration after concentrated twice. Concentration of Anthocyanin extract was also reported by Nayak and Rastogi [31] using FO process. The FO produced Anthocyanin extract with a final concentration of 2.7 g/L (from 0.05 g/L) in 18 h operation. However, they observed salt back diffusion during the process. Nayak et al. [25] also successfully demonstrated Anthocyanin concentration in Beetroot and grape juices with final concentrations of up to 2.91 g/L (57 fold) and 715.6 mg/L (6.8 fold). Not only able to obtain a high concentration, the antioxidant properties of Anthocyanin in Jaboticaba juice were preserved [33]. Wrolstad et al. [35] studied the performance of FO during Raspberries concentration. They analyzed the concentrated juice and compared with the commercial one. Results of aroma and flavor analysis indicated no significant change in concentrated juice. Khongnakorn and Youravong [32] used FO for recovering protein from Tuna cooking juice. The FO could produced Tuna cooking juice with up to 9% protein content and 2.54 LMH water flux. These studies confirmed that FO can concentrate liquid food and is capable to preserve the nutraceuticals content.

Babu et al. [29] investigated Pineapple juice concentration by using mixed sucrose-NaCl as the DS. The pineapple juice was successfully concentrated up to 60 °Brix from its initial TSS of 12.4 °Brix when the DS contained 40% sucrose-12% NaCl. The mixed DS solution was used to reduce diffusion of salt to the juice thereby avoiding a salty taste. A study conducted by Garcia-Castello et al. [30] showed that FO could achieve up to 5.7 concentration factor which was higher than obtained by RO (up to 2.5). However, FO showed a lower flux than pressure-driven membrane based-processes. Therefore, FO membrane needs more improvement for achieving higher water flux and lower salt reverse diffusion.

3. Challenges in FO Operation

Despite the excellent performance of FO in dewatering liquid foods, most FO processes still show lower water flux. Even though the process uses DS with a high solute concentration, the flux is still low, e.g. below 10 LMH [37]. The low water flux will require a large membrane area which not satisfy the practical application. Furthermore, some operational problems should be overcome. FO membrane needs substantial improvement to solve reverse solute diffusion, concentration polarization, and low mechanical strength [8].

3.1. Internal concentration polarization (ICP)

The first major problem encountered in FO process in internal concentration polarization (ICP). ICP occurs due to the permeation of water from FS to DS which reduces the solute concentration of DS at the support-active layer interface. Consequently, this results in osmotic pressure reduction and thereby decreases water flux [38]. It is also accompanied by the low reverse salt diffusion from the bulk DS to the interface [39].

Developing membrane with a low ICP is needed to improve FO performance [40]. Modifying the membrane support layer should be attempted since ICP cannot be controlled by tuning FO operating condition [40]. As ICP is determined by support layer structure and hydrophilicity, extensive efforts have been devoted to improve those parameters [41–44]. Generally, structural parameter of S is used to determine the ICP tendency of the membrane. S is defined as: $S = \text{thickness} \times \text{porosity/tortuosity}$. The effect of S on water flux is shown in Figure 2. For instance, minimizing ICP can be done by fabricating a very thin support layer.
However, another problem arises, that is a weak mechanical strength. Another factor affecting ICP and membrane flux is FO operation mode. More severe ICP was found when FO was operated at AL-FS rather than AL-DS [9]. However, AL-FS experienced a higher flux stability than AL-DS [9].

Chanukya and Rastogi [34] proposed ultrasound assisted FO for reducing concentration polarization in concentration of sucrose and pectin containing solutions. Ultrasound assisted FO exhibited a higher water flux and a lower concentration polarization than the conventional FO. However, it makes the equipment more complex.

![Figure 2. Effect of structural parameter S on water flux (membrane orientation: AL-FS; DS = 2 M NaCl; data from refs. [46–53])](image)

3.2. Reverse solute diffusion from DS to FS

Another problem that should be taken into consideration is salt flux or reverse salt flux [37]. Reverse solute diffusion arises due to the high concentration different originated by a high solute concentration of DS and a lower solute concentration of FS. This problem, for example, was observed by Nayak and Rastogi during the concentration of anthocyanin extract. The diffusion of NaCl was found to be 0.21 moles m$^{-2}$ s$^{-1}$ [31]. Reverse salt diffusion will reduce product quality, especially a salty taste of fruit juice.

Water and salt fluxes of several membranes are depicted in Figure 2. Generally, a trade-off between selectivity and permeability is found in membrane [54,55]. A membrane which has high permeability and selectivity is preferable. Such membrane is needed to achieve a high productivity and to obtain a high-quality product. Therefore, the membrane should be designed to the direction of lower-right side of $J_w$ vs $J_s$ curve (Figure 2).
Figure 3. Water and reverse solute fluxes ($J_w, J_s$) of various FO membranes (FS = deionized water; DS = 2.0 mol/L NaCl; data from refs. [50,51,56–63]).

3.3. Fouling

FO offers several advantages compared to other pressure-driven membrane processes, including the low pressure requirement and the ability to treat a high solid content solution [37]. The low fouling tendency of FO is due to the low hydraulic pressure and low permeating flux resulting in a non-compacted and reversible fouling [21]. Even though the fouling tendency is lower than other pressure-driven membrane processes, fouling is still the major drawbacks in FO process [8]. For instance, during the orange peel press liquor concentration, Garcia-Castello and McCutcheon [23] found that fouling phenomena occurred in FO process. The fouling reduced water permeate flux up to 50%. Pectin was suspected as the dominant fouling contributor. Fouling phenomena in FO process was also found by Khongnakorn and Youravong. They found that protein was the suspected foulant in Tuna cooking juice [32].

Zou et al. reported that [64] more severe fouling was observed when a high concentration of DS was used. This is because a high concentration DS leads to a high permeating flux. Consequently, it will cause more rapid foulant deposition on the membrane surface. Even though FO is also hampered by fouling, the fouling structure was found to be different than those encountered in RO [19]. The fouling layer had looser structure and less compacted thus increasing the efficacy of membrane cleaning [19]. To address this issues, many novels membranes have been developed.

4. FO Membrane Modifications

Generally, two types of membrane are used for FO, i.e. asymmetric cellulose acetate and thin-film composite (TFC) polyamide membranes [65]. The second one shows a higher water flux rather than the first but more susceptible to fouling. TFC membrane is more popular than other FO membranes as it has higher permselectivity and provides more design options, in which active and support layer can be designed separately [66]. TFC membrane exhibits superior characteristics compared to other membranes, in terms of water permeability, salt rejection, mechanical strength, and chemical stability [53,54,67,68]. Most TFC membrane suffers from ICP phenomena due to reverse salt diffusion and support structure thus results in a dramatic water flux reduction. TFC comprises of active layer with a high salt rejection and support layer with high mechanical strength. A support layer with a low tortuosity and a high hydrophilicity is preferable to reduce ICP [54].
Ideally, FO membrane should have high water permeability, high solute rejection, and high mechanical strength. To achieve those properties, membrane active and support layer need to be tuned in order to minimize mass transfer limitation in the support layer and maximize the selectivity of membrane active layer [39]. The active layer is responsible for water permeating flux and reverse solute flux. Accordingly, the active layer should be selective toward water. Meanwhile, the membrane support layer should have high mechanical strength for supporting the active layer. As previously mentioned, it should be noted that the support layer also affects the water flux due to ICP phenomenon. Therefore, the support layer properties including thickness, porosity, and tortuosity should be taken into consideration in the fabrication [69].

Various support optimization strategies were reported, such as additive blending [56], chemical etching [70], in-situ mineralization [46], surface modification [71], and template assisted technique [72]. The aim was to increase membrane porosity and hydrophilicity as well as to reduce membrane tortuosity [53]. Water ($J_w$) and solute fluxes ($J_s$) of various support types are shown in Figure 4.

It was demonstrated by numerous studies that the use of nanoparticles in membrane fabrication could improve the membrane performance [73–75]. Incorporating inorganic nanoparticles into membrane support promises an effective ICP reduction. For example, Zhang et al. [53] synthesize polyvinylidene fluoride (PVDF) membrane support by introducing SiO$_2$@MWNTs as additive. The membrane showed a higher water flux and an improved selectivity. The improved selectivity was observed from a reduction of $J_s/J_v$ values from 1.10 to 0.19 g/L. Liu et al. [46] used hydrophilic mineral coating for improving hydrophilicity of polyethersulfone (PES)-based support. The pores surface of the support was coated by CaCO$_3$. The hydrophilic nature of CaCO$_3$ increased PES-based support hydrophilicity leading to the reduced ICP. The prepared TFC membrane had a water flux of 52 LMH when operated at AL-FS mode (FS = deionized water, DS = 2 M NaCl). Even though nanoparticles addition resulted in an improved membrane performance, another problem is emerged that is aggregation of nanoparticles in high concentration due to reducing miscibility [76].

Polymer blending is another facile method for improving support hydrophilicity [77,78]. Chen et al. [40] reported polymer blending method for preparing polysulfone (PSf)/sulfonated poly (ether ether ketone) (SPEEK) support. PSf/SPPEK showed a high hydrophilicity and open pore structure. The blended polymer resulted in a higher water flux (28.3 LMH). The same method was applied by Zhang et al. [79] in fabrication of PSf support. Disulfonated poly (arylene ether sulfone) hydrophilic-hydrophobic multiblock copolymer (BPSH100-BPS0) and PSf were blended. TFC membrane with a support containing 25 wt% BPSH100-BPS0 could achieve 40.5 LMH water flux when deionized water and 2 M NaCl were used as FS and DS, respectively. The membrane also had a high water flux of 18.6 LMH during seawater desalination.

Several studies also reported that electro-spun membrane support demonstrated a low ICP with a high water flux [66]. Pan et al. [66] fabricated polyamide (PA)/polyacrylonitrile (PAN) TFC membrane. The PAN support was prepared by electrospinning method. Then, the PAN support was laminated by paper laminator. The laminated PAN support showed a high hydrophilicity with 32.3 ± 1.3° water contact angle. Moreover, the membrane could achieve water flux of 57 LMH when 2 M NaCl was used as DS. The scaffold-like structure of electrospun PAN support with interconnected pore was found to increase the support porosity thus reducing ICP phenomenon. However, electro-spun membrane is usually weak.

Chemical modification is another promising strategy for improving FO membrane performance. Chemical modifications can endow a membrane with a high hydrophilicity, anti-fouling property, and reduced ICP [8]. Guan and Wang [80] prepared cellulose triacetate membrane for FO with modified nonwoven fabric for the support. The support surface was
modified by polyvinyl alcohol (PVA) and glutaraldehyde via crosslinking process. The modification successfully increased the support hydrophilicity indicated by the reduction of water contact angle (WCA) from 116.9° to 38.5°. Meanwhile, the hydrophilicity of modified cellulose triacetate membrane was increased shown by WCA reduction from 114.1° to 55.8°. The modified membrane had water flux of 55 LMH and structural parameter of 93.65 μm. However, chemical modification still result in a trade-off between membrane performance and mechanical strength [8].

Modifying membrane surface or surface modification is one of the most used method in fouling control strategies [81]. For instance, Zhang et al. [65] modified a polyamide membrane by in-situ surface grafting of amine-terminated sulfonated poly(arylene ether sulfone) (NH2-BPSH100). The modified membrane showed an improved antifouling property during a filtration of soybean oil/water emulsion (40 g/L). Membrane flux could be easily recovered by a simple hydraulic flushing resulting in a higher flux recovery factor (69.8%) rather than the unmodified membrane (11%). Park et al. [82] introduced a simple method for increasing the hydrophilicity of PVDF nanofiber support, that is by dip coating of the membrane support into polyvinyl alcohol (PVA) solution. The modified TFC membrane showed an excellent water flux of 34.2 LMH with 1 M NaCl and deionized water as DS and FS, respectively.

Kang et al. [84] prepared a regenerable FO membrane with multiple polyethylenimine (PEI) and poly(acrylic acid) (PAA) bilayers as the active layer. The fouled membrane can be regenerated by disassembling the PEI-PAA bilayers using acid solution followed by coating the new PEI-PAA bilayers on a polydopamine-functionalized polysulfone support. This approach may reduce membrane replacement cost since it only requires to replace the active layer.

To solve the issue of ICP as well as a weak mechanical strength, Li et al. [38] developed a self-standing FO membrane without support layer. The membrane was synthesized from COOH derived polyoxadiazole copolymer. The membrane has a few micron thickness (5-15 μm). The membrane showed a high Na2SO4 rejection and an acceptable water permeability.
(8.37 × 10⁻⁷ LMH bar⁻¹). This result shows the potential of a support-free membrane for eliminating ICP.

Graphene oxide (GO) membranes are considered as a potential next-generation membrane as they have ultra-thin thickness, tailorable pore size, and superior water permeability [85]. Those advantages, for example, have been utilized by Yang et al. [85] to prepare FO membrane. They fabricated polydopamine coated reduced GO membrane. They found that the membrane exhibited a high water flux 36.6 LMH with relatively low reverse solute flux 0.042 mol L⁻¹ m⁻² and high salt rejection 92.0% [85].

5. Conclusions and Future Perspectives

Maintaining nutritional value is quite challenging in liquid food concentration, especially for thermally based processes, since the nutraceutical compounds are heat sensitive. Among the alternative processes, FO offers several attractive features allowing to address the drawbacks of the thermally based processes. In addition to its ability in producing a final product with a high solid concentration, FO can retain the bioactivity of nutraceuticals due to its mild operating condition. Despite the advantages, FO faces several problems that hamper the application, namely concentration polarization, fouling, and reverse salt diffusion [5]. Membrane properties play the significant role in controlling those problems. Therefore, many efforts focus on developing membrane which can alleviate those issues. Despite the advancement in FO membrane preparation, most of the prepared membranes are characterized by deionized water as FS [24]. For practical application, the developed membrane should be tested in a real solution.

6. References

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