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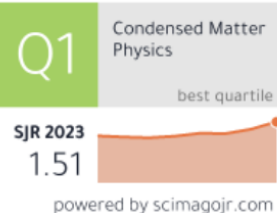
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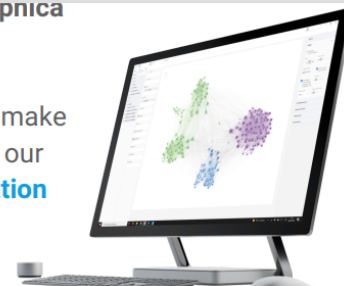
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Title: Hydriodic iodide and iodine permeation characteristics of fluoropolymers as a lining material

Article Type: Short Communication

Section/Category: Purification / Separation / Membranes

Keywords: Iodine-Sulfur process, hydrogen, lining material, fluoropolymer, permeation

Abstract: The thermochemical water-splitting iodine-sulfur (IS) process requires corrosion-resistant materials owing to usage of corrosive fluids, such as a mixture of HI-I₂-H₂O. Fluoropolymers, such as PTFE and PFA, are adaptable as lining materials for protecting plant components. However, there has been a concern: PTFE and PFA have the ability to permeate various permeants. From the viewpoint of corrosion of the base material, the permeation characteristics of HI and I₂ should be evaluated to improve the integrity of the IS process. In this study, permeation tests on PTFE and PFA membranes were performed to measure the permeated fluxes of HI and I₂, and the effects of the operating conditions on them were investigated. The introduction of a permeability parameter could be successful for normalizing the permeated fluxes for a specific membrane thickness and a vapor pressure. Then, the empirical formula of the permeability was given as an Arrhenius-type equation to use as a plant design. Finally, based on the results, the proper conditions for design of a lining material for the inhibition of HI and I₂ permeation are summarized.



Title: Hydriodic iodide and iodine permeation characteristics of fluoropolymers as a lining material

Author: Nobuyuki Tanaka, Hiroki Noguchi, Yu Kamiji, Hiroaki Takegami, Shinji Kubo

<Abstract>

The thermochemical water-splitting iodine–sulfur (IS) process requires corrosion-resistant materials owing to usage of corrosive fluids, such as a mixture of HI-I₂-H₂O. Fluoropolymers, such as PTFE and PFA, are adaptable as lining materials for protecting plant components. However, there has been a concern: PTFE and PFA have the ability to permeate various permeants. From the viewpoint of corrosion of the base material, the permeation characteristics of HI and I₂ should be evaluated to improve the integrity of the IS process. In this study, permeation tests on PTFE and PFA membranes were performed to measure the permeated fluxes of HI and I₂, and the effects of the operating conditions on them were investigated. The introduction of a permeability parameter could be successful for normalizing the permeated fluxes for a specific membrane thickness and a vapor pressure. Then, the empirical formula of the permeability was given as an Arrhenius-type equation to use as a plant design. Finally, based on the results, the proper conditions for design of a lining material for the inhibition of HI and I₂ permeation are summarized.

Keywords: Iodine–Sulfur process, hydrogen, lining material, fluoropolymer, permeation

1. Introduction

Fluoropolymers, such as PTFE (PolyTetraFluoroEthylene) and PFA (PerFluoroAlcoxy), have been applied to line plant components, such as pipes and containers, in a variety of chemical process owing to their superior chemical inertness [1-3]. Our group has developed a certain chemical process: the thermochemical water-splitting iodine–sulfur (IS) process. The IS process is one of the most promising hydrogen (H₂) production methods because it has considerable potential to provide carbon-free H₂ on a large-scale from nuclear and solar energy [4-6]. The IS process involves the following three chemical reactions:



Through reactions (1) to (3), the IS process enables us to obtain H₂ and oxygen (O₂) by splitting water. To proceed with the reactions, corrosive fluids, such as sulfuric acid (H₂SO₄) and hydriodic acid (HI) including iodine (I₂), should flow [4] so that the corrosion-resistant materials are required to protect the components in the IS process. To meet this demand, for the IS process, PTFE- and



PTA-lined materials have been adopted [7].

Generally, PTFE and PFA have a great chemical stability; however, they easily permeate many kinds of permeants [1,8]. When PTFE and PFA are used as lining materials, this unwanted permeation characteristic may cause issues during use, such as the corrosion of base materials and delamination of the liner [1,9]. Unfortunately, the IS process is no exception to this problem. It is empirically well-known that HI and I₂, which are the main corrosive reactants of the IS process, permeate through PTFE and PFA, even though barely. The permeation, although not detrimental in the short term [7], may be of concern during long-term operation on a practical level. For example, the permeated HI and I₂ may corrode component materials more than predicted, leading to a short lifetime, or the process may be more costly than necessary to maintain. Therefore, concerns regarding the permeation of HI and I₂ must be overcome to manage the integrity of the IS process so that component materials have a long life time and low cost. To dispel these concerns, permeation data on HI and I₂ through PTFE and PFA have become important, and the effect of permeation should be quantitatively evaluated. However, quantitative data has not been reported thus far.

In this study, permeation tests on PFA and PTFE to measure the permeated flux and permeability of HI and I₂ were performed. The sequential tests enabled us to provide effective data for a plant design. The effects of the operating temperature and types of membrane on the permeation characteristics were investigated. Consequently, the proper conditions for the inhibition of permeation during the design of lining materials are summarized.

2. Experimental methods and analysis

2.1 Chemicals and membranes

Hydriodic acid (HI, 55–58 wt.%, Kanto Chemical Co., Inc.) and iodine (I₂, >99.8 wt.%, Kanto Chemical Co., Inc.) were used as received to prepare the HI-I₂-H₂O mixture (HIx solution). Sodium thiosulfate solution (0.1 M Na₂S₂O₃, Kanto Chemical Co., Inc., Japan), a sodium hydroxide solution (0.1 M NaOH, Kanto Chemical Co., Inc.), and potassium iodide powder (KI, >99.7%, Kanto Chemical Co., Inc.) were utilized to analyze the composition of the HIx solution using an automatic potentiometric titrator (COM-2500, Hiranuma Sangyo Co., Ltd.).

PTFE and PFA membranes (FLON INDUSTRY) were selected as the test membranes. The thicknesses of PFA were 25 and 250 μm, and that of PTFE was 50 μm.

2.2 Experimental procedure for the permeation test

Permeation tests were performed to measure the permeation amount of HI and I₂ through PTFE and PFA membranes. A membrane with 45 cm² of effective membrane area was sandwiched in a test cell made of glass and set parallel to the ground. To the bottom side of the cell, 100 mL of the prepared HIx solution (the concentration was HI = 12 mol/kg and I₂ = 14 mol/kg) was added, and

the vapor of this solution contacted the membrane. In the top side, nitrogen (N₂) gas was purged at a flow rate of 20 mL/min to expel permeants from the test cell. The cell was heated by a mantle heater and kept constant. Test temperatures were varied in the range of 30 to 85°C. The purged N₂ gas was bubbled into a water trap to dissolve HI and I₂ gas permeated through the tested membrane within 6 h after heating up. Then, the solution in the water trap was sampled, and total amounts of the permeated HI and I₂, m_i [mol], were measured by titration.

2.3 Analysis of the flux and permeability coefficient

The permeated flux of component i , J_i [mol/m²·s], through a membrane can be described as [10-12]:

$$J_i = -D_i \frac{\partial c_i}{\partial x} \quad (4)$$

where x [m], D_i [m²/s], and c_i [mol/m³] represent the direction of membrane thickness, the diffusion coefficient, and the concentration of component i , respectively. In the steady state, diffusion flow is constant. Assuming that D_i is independent of c_i , Eq. (4) can be integrated to give

$$J_i = D_i \frac{(c_{i,f} - c_{i,p})}{L} \quad (5)$$

where $c_{i,f}$, $c_{i,p}$ [mol/m³], and L [m] represent the concentration of component i at the feed side and permeate side and the membrane thickness, respectively. According to Henry's law [10-12], c_i is proportional to the partial vapor pressure of component i , p_i [atm],

$$c_i = S_i p_i \quad (6)$$

where S_i [mol/m³·atm] represents the solubility coefficient of component i . The vapor pressure at the permeate side can be regarded as zero owing to N₂ gas purging. The product of $D_i S_i$ is called the permeability coefficient, P_i [mol/ m·atm·s] [10] so that Eq. (5) can be transcribed as

$$P_i = J_i \frac{L}{p_{i,f}} \quad (7)$$

This equation means that P_i can be determined by measuring J_i . In our study, p_i values were estimated from the database of the OLI Engine licensed by OLI systems, Inc.

3. Result and discussion

Figure 1 shows the J_{HI} and J_{I_2} values through the PTFE and PFA membranes at several temperatures. All the J_i values increased with an increase in temperature. From the viewpoint of corrosion, the permeation of HI and I_2 through membranes is an undesirable phenomenon. Therefore, a higher temperature environment could disadvantage membranes for permeation. Moreover, the effect of membrane thickness was investigated by using PFA membranes that were 25 and 250 μm . Lower J_{HI} and J_{I_2} values were observed when using the thicker PFA membrane. The results could be rationalized because J_i is inversely proportional to thickness, as given by Eq. (5). This suggests that the usage of a thicker membrane could be effective to inhibit HI and I_2 permeation.

Here, we investigate how J_i differs between the PFA and PTFE membranes. Equation (5) shows that J_i depends on the membrane thickness and vapor pressure of each permeant, indicating that the membrane and molecule properties could concurrently affect the permeation characteristics. Therefore, it was difficult to individually evaluate the effect of each membrane on the permeation characteristics of HI and I_2 only from the results shown in Figure 1. Thus, parameter P_i described in Eq. (7) is introduced. P_i is normalized to the membrane thickness and vapor pressure; therefore, P_i can serve as an intrinsic indicator of membranes for the i component to evaluate the permeation characteristics of each molecule. Figure 2 plots the P_i values for the PTFE and PFA membranes as a function of temperature. These plots were calculated from the measured J_i shown in Figure 1. Of note, the P_i values of PTA and PTFE membranes have been reported for various molecules by various researchers [1-3]. For example, S. Moon et al. [2] measured the P_i of hydrogen chloride for PFA membrane at 25°C to be 3.8×10^{-10} – 1.2×10^{-9} mol/ m·atm·s. This suggests that HI and I_2 could show a similar P_i value for the tested membranes to those of other molecules.

As shown in Figure 2, all the P_i values increased with an increase in temperature. The temperature dependence had a linear relationship on the logarithmic scale to the plots of each permeant molecule for each membrane. Additionally, the P_i values for the PFA membrane were on one straight line for both HI and I_2 despite the results for different thicknesses, indicating that the effect of membrane thickness was normalized by the P_i indicator. This supports that P_i is appropriate for evaluating the peculiarity of the membrane itself, excluding the effect of membrane size. The temperature dependence of P_i is assumed to be expressed by the following Arrhenius-type formula [10]:

$$P_i = P_{0,i} \exp\left(-\frac{E_i}{RT}\right) \quad (8)$$

where E_i [J/mol] and $P_{0,i}$ [mol/ m·atm·s] represent the activation energy and pre-exponential factor of component i , respectively. In Figure 2, the fitted lines for Eq. (8) can accurately reproduce the experimental values within the experimental errors. Table 1 lists the calculated fitting parameters.

Both the P_{HI} and P_{I_2} values increased with temperature, and the P_{HI} values of both

membranes were higher than the P_{I_2} values within the range of our measured conditions. As shown in Table 1, E_{I_2} was higher than E_{HI} for both membranes, suggesting that I_2 needs considerable activation energy to permeate the membrane compared with the HI molecule. Considering that the molecular diameter of I_2 is larger than HI [13] as well, the permeation resistance of I_2 may be larger than that of HI. Interestingly, the differences between P_{HI} and P_{I_2} were smaller than those of J_{HI} and J_{I_2} shown in Figure 1, irrespective of the types of membranes. According to Eq. (7), membrane thickness can influence the respective molecules similarly; therefore, the thickness is independent of the difference between P_{HI} and P_{I_2} . Consequently, the influence of J_i was mainly derived from another parameter: the vapor pressures of HI and I_2 .

Regarding the difference between membranes, both the P_{HI} and P_{I_2} values for PFA were smaller than those for PTFE, indicating that PFA has a higher suppressibility of permeation than PTFE. However, as shown in Table 1, the E_i values of PTFE were higher than those of PFA, indicating that PTFE has a higher energy barrier for permeation than PFA. Therefore, HI and I_2 had difficulty permeating PTFE compared with PFA. This is contrary to the fact that the P_i values of PFA were lower than those of PTFE. An additional view is necessary to explain this. Specifically, the affinity of HI and I_2 to the membranes should be considered. Unfortunately, based only on the obtained results, it is difficult to provide a more detailed explanation regarding the difference between the membranes. However, it is important to obtain permeation characteristics data. However, because a detailed discussion of the permeation phenomena was not the focus of this study, it is postponed to future research.

Practically, the allowable J_{HI} and J_{I_2} should be quantitatively determined from many factors that consider the lifetime of materials, cost of the plant, etc., which depend on the usage environment of plants, such as the operating temperature and fluid concentration. In the IS process, the permeation of HI and I_2 through lining material is undesirable owing to their corrosive properties; therefore, their lower permeation is required. In the following, methods to inhibit permeation will be summarized based on the measured results. The permeation can be controlled based on the operating conditions and materials. First, the operating temperature in the usage environment was evaluated. As discussed above, a lower temperature allows J_i to decrease because decreasing the temperature can reduce not only the P_i value itself but also p_i . Thus, a decrease in the operating temperature plays an important role in inhibiting permeation of HI and I_2 , whereas there are some cases when a higher temperature is the preferable condition for plant operations. It is known that the operating temperature considerably affects the thermal efficiency of hydrogen production [6]. Therefore, the lining material should be utilized in a low-temperature environment within limits to achieve a high thermal efficiency.

Regarding materials, PFA is more suitable as a lining material than PTFE because the J_i of PFA was lower under the same conditions. Besides the operating conditions, J_i only depends on

the membrane thickness according to Eq. (5). Thus, once the material and the process conditions, such as the operating temperature, are determined, J_i can only be controlled by the membrane thickness. In addition, Eq. (5) shows that the membrane thickness is inversely proportional to the permeated flux, indicating that a thicker membrane prevents HI and I₂ from permeating. However, an increase in thickness may lead to another issue, such as component material cost increases. Therefore, the lining material should be designed with the minimal thickness to inhibit the permeation of HI and I₂.

4. Conclusion

In this study, the characteristics of HI and I₂ permeation through PFA and PTFE membranes were investigated. The tested membranes are essential lining materials for anti-corrosion in the IS process, and quantitative permeation data of HI and I₂ are required to evaluate the integrity of component materials. The newly added data from our study can help to improve the integrity of the IS process. The experiments showed that the permeated fluxes of HI and I₂ depend on the operating temperature and membrane thickness. Additionally, the permeability, which can normalize the effects of the membrane thickness and molecule vapor pressure, was introduced to evaluate the permeation characteristics of HI and I₂. The permeability of HI was higher than that of I₂, and PFA was difficult to permeate by HI and I₂ compared with the PTFE membrane under our experimental conditions. Additionally, the temperature dependence of the permeability was reproduced by an Arrhenius-type experimental formula. The fitting parameters are provided so that the formula can be used for process design.

For inhibition of HI and I₂ permeation, suitable lining materials and operating conditions for the IS process were evaluated. The operating temperature considerably affected the permeation characteristics as well as the process efficiency. A process with a lower temperature should be designed that has a small effect on process efficiency. PFA may be preferable to PTFE as a lining material. Although the material cost should be considered, a thicker membrane may be more beneficial for inhibiting permeation.

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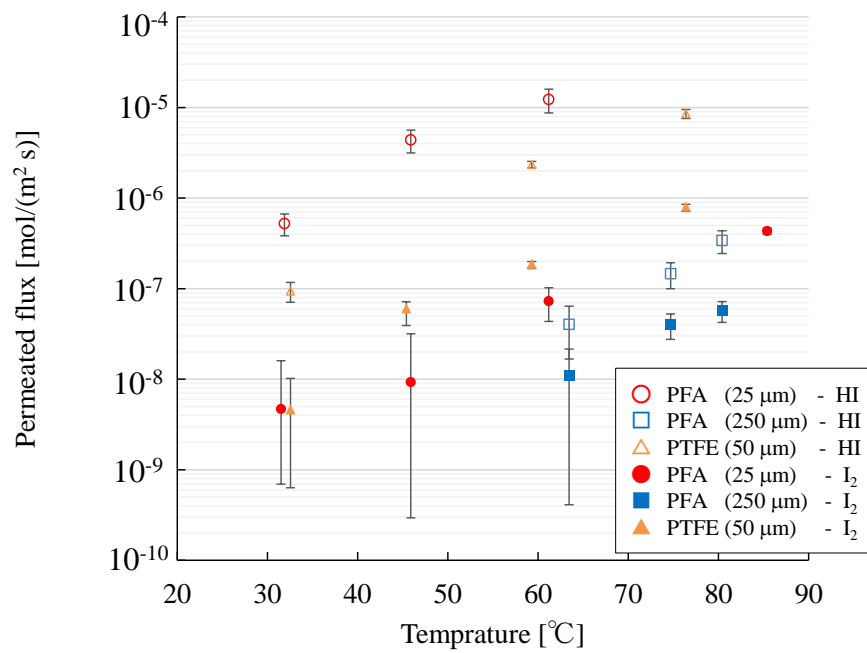


Fig. 1. Permeated flux of HI and I₂ through PFA and PTFE membranes as a function of operating temperature

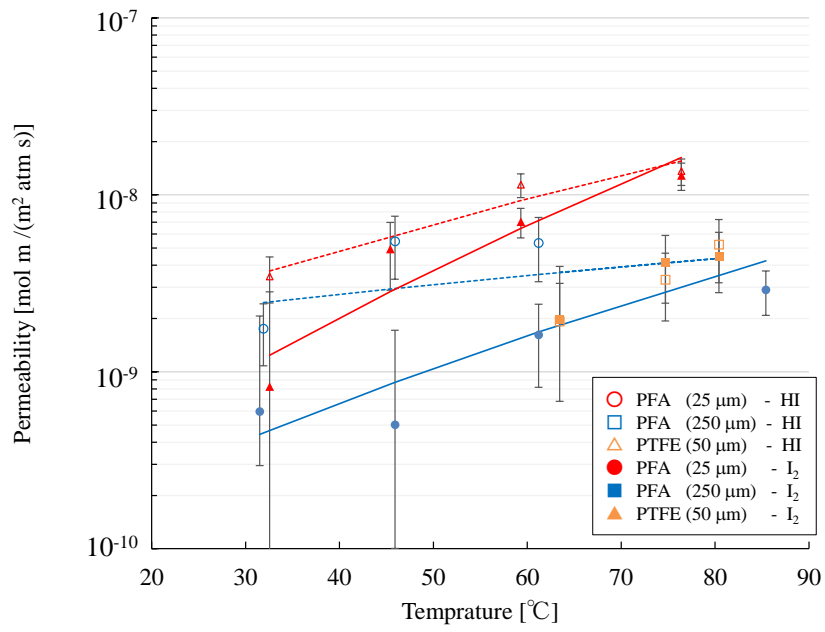


Fig. 2. Permeability of HI and I₂ against PFA and PTFE membranes as a function of operating temperature (plots: experimental data, lines: fitting curve based on the Arrhenius-type equation)

Table 1. List of parameters fitted by the Arrhenius-type equation for the permeability

membrane	molecule	$P_{0,i}$ [mol · m/m ² · atm · s]	E_i [kJ/mol]
PFA	HI	1.6×10^{-7}	10.6
	I ₂	1.5×10^{-3}	38.0
PTFE	HI	3.3×10^{-4}	29.0
	I ₂	1.0	52.1