Preparation, characterization, and performance evaluation of TiO$_2$-ZrO$_2$ membranes for nanofiltration

(ナノ濾過 TiO$_2$-ZrO$_2$膜の作製、特性評価および透過特性の評価)

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The overall objective of this dissertation research is to develop TiO$_2$-ZrO$_2$ membranes for nanofiltration. Among ceramic membrane materials, TiO$_2$ and ZrO$_2$ are widely known as superior inorganic materials in terms of chemical stability towards aqueous solutions over a wide range of pHs. However, it is difficult to control and achieve TiO$_2$ and ZrO$_2$ membranes with crack-free top layers and small pore sizes in the nanofiltration range due to their low-phase transformation temperature. Generally, the crystallization temperature of the pure oxides can be increased by the addition the other oxides, which has been proven by increasing crystallization temperature of TiO$_2$-ZrO$_2$ composite materials over their crystallization temperature of the pure oxides. Since it is believed that the membrane performance is controlled by pore structure of the membranes, controlling the sol size and operating condition during preparation of the membrane is crucial process. Therefore, this research has been focused on the preparation, characterization, and evaluation performance of TiO$_2$-ZrO$_2$ composite membranes for nanofiltration, which are expected to possess high thermal and chemical stability under harsh environments. In addition, no organic additive was used in the TiO$_2$-ZrO$_2$ sols, which provide greater levels of green processing. Moreover, nanofiltration experiments in aqueous solutions were carried out to evaluate the water permeability and separation performance of the membranes. In this study, the TiO$_2$-ZrO$_2$ nanofiltration membranes were successfully prepared by using sol-gel method. The structure and separation performance of the membranes were successfully controlled by the use different size of the sols, by manipulating the firing temperature, and by the use of different Ti/Zr ratios. TiO$_2$-ZrO$_2$ membranes with Ti/Zr ratio of 9/1, 7/3, and 5/5 fired at 400 °C showed a relatively low molecular weight cut-off and a moderate water permeability, which suggested that these membranes would be good for nanofiltration. In addition, hydrothermal stability tests and nanofiltration performance at temperatures ranging from 25 to 85°C revealed that TiO$_2$-ZrO$_2$ (5/5) membranes fired at 200, 400, and 550 °C possessed excellent hydrothermal stability high temperatures. Thus, the present study will provide a general strategy for development of high-performance membranes for use in nanofiltration, especially for filtration applications under harsh environments.