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二维材料基疏松纳滤膜制备及其有机小分子/二价盐分离性能研究

摘要

轻工行业的快速发展使含染料、氨基酸及抗生素等有机小分子的高盐废水排放不断增加。实现有机小分子与无机盐（二价及高价盐）的高效分离，对废水资源化及零排放具有重要意义。本研究聚焦高性能疏松纳滤（LNF）膜的构建，以缓解“渗透-分离”的“trade-off”效应。基于界面聚合优化，系统考察二维材料氧化石墨烯（GO）与二硫化钼（MoS_2）插层对膜结构与性能的影响。通过正交试验确定 PVDF 基膜最优界面聚合条件：PIP 浓度 4 w/v.%、停留 300 s，TMC 浓度 0.05 w/v.%、停留 90 s，热处理 30 min，可获得稳定 NF 膜。在 GO 改性中，对比铸膜液添加、插层及水相添加三种方式，发现以多巴胺（DA）为桥连的插层结构最优，制得 PVDF/PDA-GO LNF 膜。该膜通量为 10.45 L/(m^2·h·bar)，PEG2000 截留率 76.05%，Mg2SO4 截留率 4.88%。机理分析表明分离主要受空间位阻与 Donnan 效应协同控制。进一步以低成本 MoS_2 替代 GO，通过真空抽滤结合原位限域界面聚合，构建 H-PVDF/MoS2 LNF 膜。该膜纯水通量达 830.42 L/(m^2·h·bar)，刚果红/Mg2SO4 分离系数达 52.01，显著削弱 trade-off。其高通量源于亲水性与较大孔结构，而负电表面及 0.63 nm 层间距实现高效选择性分离。同时，膜在酸性条件下通量恢复率达 95.92%，并可在 0.08 MPa 低压下稳定运行，展现出良好的工业应用潜力。

关键词

有机小分子；无机盐；界面聚合；疏松纳滤膜；机理探究

Abstract

The rapid development of the light industry has led to an increasing discharge of high-salinity wastewater containing small organic molecules such as dyes, amino acids, and antibiotics. Achieving efficient separation of small organic molecules and inorganic salts (divalent and multivalent salts) is of great significance for wastewater resource utilization and zero discharge. This study focuses on the construction of high-performance loose nanofiltration (LNF) membranes to alleviate the “permeation-separation”trade-off effect. Based on interfacial polymerization optimization, the effect of intercalation of two-dimensional materials—graphene oxide (GO) and molybdenum disulfide (MoS_2)—on membrane structure and performance was systematically investigated. Through orthogonal experiments, the optimal interfacial polymerization conditions for the PVDF base membrane were determined: PIP concentration 4 w/v.%, standing time 300 s, TMC concentration 0.05 w/v.%, standing time 90 s, and thermal treatment for 30 min, which can yield a stable NF membrane. In GO modification, three methods—addition into casting solution, intercalation, and addition into aqueous phase—were compared, and the intercalated structure bridged with dopamine (DA) was found to be the best, resulting in the PVDF/PDA-GO LNF membrane. This membrane exhibits a flux of 10.45 L/(m^2·h·bar), PEG2000 rejection of 76.05%, and Mg2SO4 rejection of 4.88%. Mechanism analysis indicates that the separation is mainly controlled synergistically by size exclusion and the Donnan effect. Furthermore, using low-cost MoS2 to replace GO and combining vacuum filtration with in-situ confined interfacial polymerization, an H-PVDF/MoS2 LNF membrane was constructed. This membrane achieves a pure water flux of 830.42 L/(m^2·h·bar), a Congo red/Mg2SO$_4$ separation factor of 52.01, and significantly weakens the trade-off effect. Its high flux is attributed to hydrophilicity and a larger pore structure, while the negatively charged surface and 0.63 nm interlayer spacing enable efficient selective separation. At the same time, the membrane achieves a flux recovery rate of 95.92% under acidic conditions and can operate stably under low pressure (0.08 MPa), demonstrating good potential for industrial application.

Keywords

small organic molecules, inorganic salts, interfacial polymerization, loose nanofiltration membranes, mechanism study

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