

辐射诱导有机-无机杂化材料的制备与性能研究进展

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摘要 有机-无机杂化材料通过有机与无机组分的协同作用克服了单一材料的性能局限,实现了材料的高性能化和多功能化,但在制备过程中仍存在组分混合不均、孔隙率下降及相容性差等难题。辐射诱导技术通过调控能量传递与反应路径,为解决这些问题提供了有效途径。本文综述了辐射诱导法制备有机-无机杂化材料的研究进展:直接法利用 γ 射线、电子束等辐射源激活活性粒子(自由基、离子),驱动化学键重组,实现常温常压下快速合成,如Ag/PVA杂化水凝胶的绿色制备及ZIF-8@ZnO异质结构的光催化性能提升;间接法通过辐射接枝在有机基材表面引入功能位点(如羧基、氨基),促进无机组分原位生长,解决界面结合难题,如MOF固定化尼龙织物及(222)取向的NH₂-ZIF-8@HF气体分离膜。辐射诱导材料在催化(硝基芳烃降解速率提升近100倍)、分离(油水分离通量达16.6 L/(m²·h·kPa))、电化学(柔性传感器耐久性增强)及机械性能(CNT复合材料强度提高至1.89 GPa/(g·cm³))方面表现突出。该技术为高性能杂化材料的设计与规模化制备提供了新思路。

关键词 辐射, 有机-无机杂化材料, 制备, 性能

中图分类号 TL13

DOI: 10.11889/j.1000-3436.2025-0047

CSTR: 32195.14.j.JRRRP.1000-3436.2025-0047

引用该文:

郭正纲, 田坤遥, 郑红星, 等. 辐射诱导有机-无机杂化材料的制备与性能研究进展[J]. 辐射研究与辐射工艺学报, XXXX, XX(XX): XXXXXX. DOI: 10.11889/j.1000-3436.2025-0047.

GUO Zhenggang, TIAN Kunyao, ZHENG Hongxing, *et al.* Research progress on the preparation and performance of radiation-induced organic-inorganic hybrid materials[J]. Journal of Radiation Research and Radiation Processing, XXXX, XX(XX): XXXXXX. DOI: 10.11889/j.1000-3436.2025-0047.



基金资助: 中国科学院青年创新促进会(E2202005)资助

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收稿日期: 初稿 2025-06-04; 修回 2025-08-20

Supported by Youth Innovation Promotion Association CAS (E2202005)

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Received 04 June 2025; accepted 20 August 2025

Research progress on the preparation and performance of radiation-induced organic-inorganic hybrid materials

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ABSTRACT Organic-inorganic hybrid materials overcome the limitations of single materials via the synergistic effect of organic and inorganic constituents, achieving high performance and multi-functionality. However, there are still challenges during preparation of organic-inorganic hybrid materials, such as non-uniform distribution of components, reduced porosity, and poor interfacial compatibility. Radiation-induced synthesis provides an effective way to address these challenges by tuning energy transfer and reaction pathways. This review summarizes recent advancements in radiation-induced synthesis techniques for organic-inorganic hybrid materials. The direct method employs radiation sources (e. g., γ -rays and electron beams) to generate reactive species (radicals and ions), facilitating the recombination of chemical bonds and allowing rapid synthesis under ambient conditions. Representative examples include the eco-friendly synthesis of Ag/PVA hybrid hydrogels and the enhancement of the photocatalytic performance by ZIF-8@ZnO heterostructures. The indirect method introduce functional groups (e.g., carboxyl and amino groups) onto organic substrates by radiation grafting, promoting in situ growth of inorganic components and solving the problem of interfacial compatibility, as demonstrated in MOF-immobilized nylon fabrics and (222)-oriented NH₂-ZIF-8@HF gas separation membranes. Hybrid materials prepared by radiation induced method exhibit exceptional performance across diverse fields, including catalysis (e.g., the degradation rate of nitroaromatics has been enhanced by nearly 100-fold), separation (oil-water separation flux reaches 16.6 L/(m²·h·kPa)), electrochemistry (the durability of flexible sensors has been improved), and mechanical properties (the strength of CNT composites has been enhanced to 1.89 GPa/(g·cm³)). This technique provides new ideas for the design and large-scale preparation of high-performance hybrid materials.

KEYWORDS Radiation, Organic-inorganic hybrid materials, Preparation, Performance

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有机-无机杂化材料是有机和无机组分通过化学键(共价键、离子键、配位键等)或物理作用(氢键、范德华力等)相互协同形成的具有独特结构和性能的复合材料^[1-2]。相比单一种类材料,有机-无机杂化材料具有可调的结构和性能,可以充分发挥有机、无机材料各自的优势,克服单一种类材料在制备与应用中的问题,从而实现材料的高性能化和多功能化^[3-5]。现有有机-无机杂化材料的制备方法包含共混法^[6]、原位聚合法^[7]、溶胶-凝胶法^[8]、插层法^[9]、涂膜法^[10]、分散法^[11]、分子自组装法^[12]等,在制备杂化材料的过程中通常存在有机-无机组分相容性差、制备工艺复杂等问题^[13]。这些问题降低了材料的机械强度和稳定性,也限

制了有机-无机杂化材料的规模化生产。辐射诱导技术为解决这些问题提供了有效途径,它能够通过改变反应过程中能量吸收和传递的过程,降低化学键形成的活化能垒,激活反应物形成自由基,从而改变反应机理^[14-16],达到减少化学试剂使用以及室温快速合成等目的。辐射诱导技术还可通过协同调控材料表面接枝/复合过程、纳米粒子的尺寸/成分/空间分布^[17-18],以及诱导缺陷驱动相变等方式^[19-21],同步完成杂化材料的“合成-活化”和“结构-功能”调控^[22]。它通过赋予材料可定制的界面特性、优异的分散性、多功能集成性、新颖的电/磁/催化拓扑结构,使得所制备的杂化材料在稳定性、机械性能、电化学性能和催化活性等方面均展现

出显著优势。这些特性使得辐射诱导技术在诸多前沿领域具有重要应用价值，如辐射合成光响应金属有机骨架(MOF)材料^[23-24]、高性能电催化剂^[25-26]、金属前药与靶向放化疗多功能平台^[27]、高效光催化材料^[28-29]等。

过往的研究报导了辐射诱导技术在不同杂化材料制备中的应用及优势，基于此，本文对辐射诱导有机-无机杂化材料的制备与性能相关的工作进行综述，将辐射诱导有机-无机杂化材料的制备方法分为直接法和间接法进行分析，并对辐射诱导法制备的有机-无机杂化材料的优异性能进行总结，旨在通过对相关工作的综述，为未来高

性能有机-无机杂化材料的设计、开发提供参考。

1 辐射诱导有机-无机杂化材料的直接制备

辐射诱导杂化材料的直接制备技术(直接法)是指辐射技术直接参与杂化材料的合成。相较于传统的杂化材料制备方法，如以热致溶剂挥发为核心的溶剂热法、以湿法溶剂交换为核心的原位合成法等，直接法很好地解决了在制备过程中所必须进行的复杂的合成步骤和长时间高温反应的严苛要求，成功实现了可以仅依靠辐射作用，在常温常压下合成杂化材料的可能(图1)^[17,30-31]。

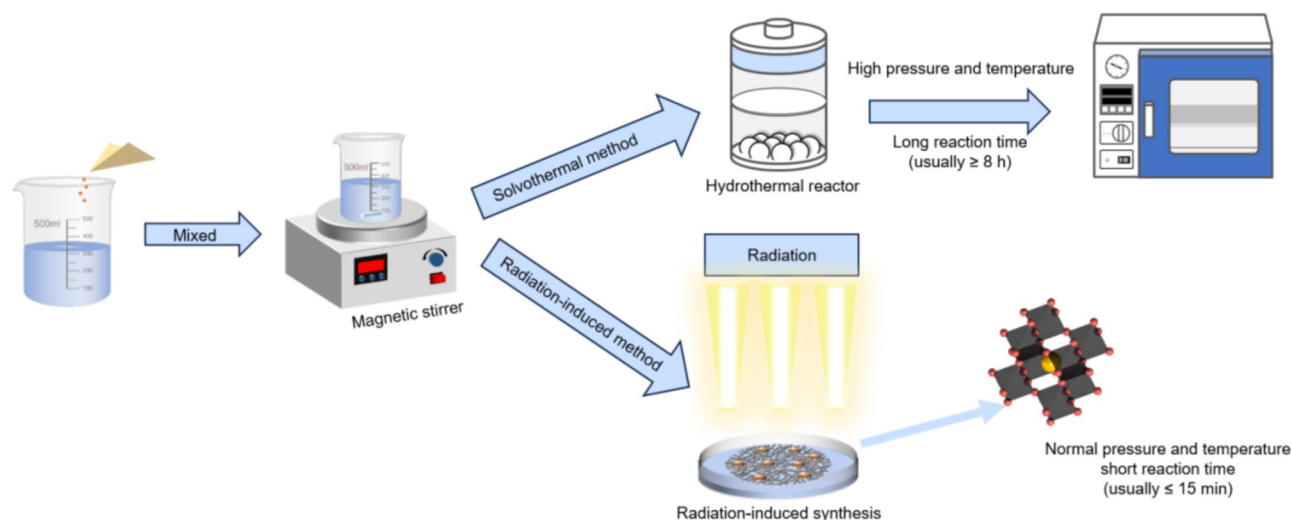


图1 溶剂热法和辐射诱导法制备杂化材料步骤

Fig.1 Preparation steps of hybrid materials by solvothermal method and radiation-induced method

1.1 直接法的机理

直接法的机理是利用辐射源(γ 射线、电子束、紫外光等)诱导反应物发生原子/分子电离、电子能级跃迁、键断裂、分子振动以及旋转跃迁等，生成具有高能量和高活性的离子、激发分子和次级电子。高活性离子通过离子-分子反应、电子俘获及中和反应生成活性自由基；激发态分子经解离、系间窜越等途径转化为自由基；高动能电子通过非弹性碰撞进一步诱发电离或激发(图2)。这些活性粒子(自由基、激发态物种)驱动分子内化学键断裂/重组与结构异构化，并在界面处促进原子扩散、吸附位点重构及共价键形成。辐射过程通过提供活化能并生成高活性反应中间体，改变反应路径，显著降低表观活化能垒^[14]。

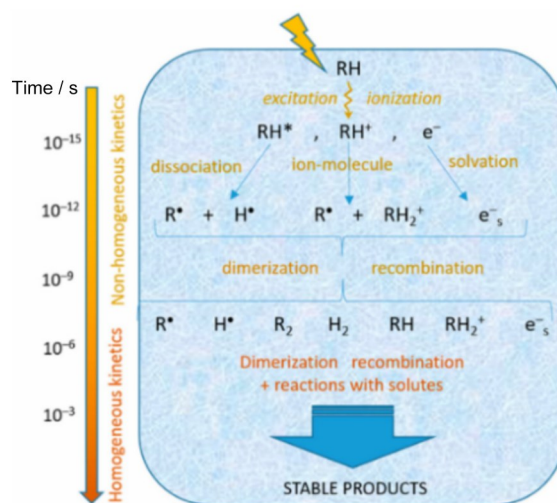


图2 电离辐射对代表性分子的辐射效应示意图^[14]

Fig.2 Schematic diagram of the radiation effects of ionizing radiation on representative molecules^[14]

1.2 直接法在有机-无机杂化材料制备中的应用

直接法可以有效减少合成过程中化学试剂的使用^[32-34],例如Chahal等采用高能射线(^{60}Co 源,5~50 kGy)同步实现聚合物交联与 Ag^+ 离子还原的绿色合成策略制备了Ag/PVA杂化水凝胶^[35-36]。利用水分子辐解产生的强还原性物种(e_{aq}^- 、 $\cdot\text{H}$ 等)直接还原金属离子并引发PVA链自由基交联(凝胶化效率>86%),显著减少化学试剂依赖且无催化剂残留,所获得的Ag/PVA水凝胶因Ag纳米粒子诱导电荷转移络合物,使光学带隙从4.92 eV降至3.72 eV,同时提升热稳定性与抗菌性。

直接法可以实现活性组分的有效结合,避免相分离,增强杂化材料的稳定性^[37-39]。例如Li等^[40]通过创新的两步辐射合成策略,利用预先辐射制备的功能化PVP纳米凝胶作为精密模板,实现了Ag和 CeO_2 纳米粒子在凝胶网络内部的原位合成、均匀分散和尺寸控制。这种方法充分发挥了辐射合成高纯度、无需添加剂、可同步功能化和精准可控的优势,最终获得的杂化纳米材料展现出小尺寸、高分散性、宽pH稳定性以及由紧密有机-无机界面带来的潜在协同性能,为设计和制备高性能多功能纳米复合材料提供了一种有效思路。

直接辐射法通过改变反应途径而显著降低反应活化能,实现杂化材料的快速合成,并且辐射效应赋予材料独特的结构使材料具有了更优异的性能^[41-42]。如Chen等^[39]首次利用市售电子加速器的高能电子束辐射,在室温下快速(分钟级)、低能耗地合成了平均直径约300 nm的高结晶度单球形ZIF-8。相较于溶剂热法,辐射技术耗能更少(比溶剂热法低约两个数量级),合成的ZIF-8具有更高的比表面积。通过调控吸收剂,电子束在合成过程中同步蚀刻材料,在ZIF-8表面形成了分级孔结构和高度结晶的ZnO纳米颗粒,从而形成了ZIF-8@ZnO异质结构,显著增强了材料的光催化降解性能。Zhang等^[43]采用 γ 射线辐照HKUST-1的甲醇溶液,利用辐射产生的还原性自由基,在温和条件下原位实现了HKUST-1骨架的部分蚀刻(形成分级孔)和 Cu^{2+} 节点的还原,成功构建了HKUST-1@ Cu_2O 异质结构。尽管辐射蚀刻和异质结构的形成导致了比表面积的减小和孔窗增大,但由此获得的独特异质结构赋予了材料更加优异的催化还原性能(速率常数提升近两个数量级)。

2 辐射诱导有机-无机杂化材料的间接制备

辐射诱导有机-无机杂化材料的间接制备技术(间接法)指的是通过辐射诱导接枝的方式间接制备杂化材料。高能辐照作用于有机基材时可以改变其表面结构,通过接枝的方式引入无机材料的生长成核点,使其具备有机-无机杂化材料生长的条件,随后再通过原位生长法、分子自组装等方法向有机基材上引入无机组分。辐射接枝可以促使有机-无机材料之间的结合更加牢固,解决有机-无机相黏合不良的问题^[44]。而与传统的化学接枝方法相比,辐射诱导不仅方法简单、经济高效,还可以通过调整吸收剂量和剂量率实现反应过程的可控化^[45-47]。此外,辐射诱导的反应过程无需引入催化剂或引发剂,既保证了产物的纯度,还能减少对环境的污染^[48-50]。

2.1 间接法的机理

间接制备法主要是利用了电离辐射能诱导化学键断裂产生自由基的特点,来对基材进行接枝改性。电离辐射如 γ 光子、X射线和高能电子等在与物质的相互作用中会诱导其发生电离,从而形成离子并释放出高速移动的电子。其中,释放的二次电子和康普顿电子会诱导更多分子发生电离,形成的离子大部分会通过去质子化反应形成自由基^[48]。有机聚合物基材在经过辐照处理后,其表面和内部会生成均匀的自由基活性位点。其中,表面的自由基活性位点可以与特定的小分子单体反应,以实现在材料表面的均匀接枝^[51]。而内部的自由基则可以相互结合,实现聚合物分子链之间的交联反应,从而改变聚合物基材的结构和物理性质^[52]。

2.2 间接法在有机-无机杂化材料制备中的应用

辐射诱导接枝的特点使得原本相容性差的物质可以结合起来^[53-55]。Yu等^[56]报道了通过辐射诱导接枝聚合(RIGP),将MOF颗粒共价固定到尼龙织物上的方法。该团队将MIL-101的纳米颗粒加入到溶解了HEA的乙醇溶液中以制备分散体,再将分散体涂抹在尼龙织物上,随后样品在室温下经由 ^{60}Co 源照射。在辐照下,MIL-101上产生的自由基与尼龙织物上不断增长的PHEA接枝链上的自由基结合,从而终止了接枝链的传播(图3)。通过这

种方式，MIL-101 和尼龙织物通过 PHEA 接枝链的桥接完成了共价连接。

辐射诱导接枝对聚合物材料具有普适性，可以拓宽杂化材料范围^[57-59]。例如，本课题组通过辐射诱导接枝的方法在聚丙烯、聚对苯二甲酸乙二醇酯、超高分子量聚乙烯等聚合物基材的表面引入马来酸酐，并以马来酸酐水解后产生的羧基基团作为活性位点，通过溶剂热法在聚合物基材表面上原位生长 MOF，制备了多种 MOF 薄膜。该方法解决了 MOF 膜材料制备过程中存在的难以大尺寸制备、膜连续性差以及连续膜制备方法普适性

差的三点难题。通过合成后修饰的方法，制备得到了含有不同官能团的 MOF 膜材料^[60-61]，如 UiO-66-AO@NWF-g-MAH、UiO-66-NH₂@NWF-g-MAH、UiO-66-NHC(S)NHMe@NWF-g-MAH、ZIF-67-N@NWF-g-MAH、UiO-66-NH₂@UP-g-MAH 等(图4)。所制备的 MOF 基杂化膜不仅均匀连续、尺寸可调，还可以通过辐射接枝参数和溶剂热参数等实现负载量和官能团的调节，系列 MOF 膜分别在油水分离、重金属分离、染料分离等领域展现出优异的性能。这一工作推动了 MOF 膜材料制备技术的发展。

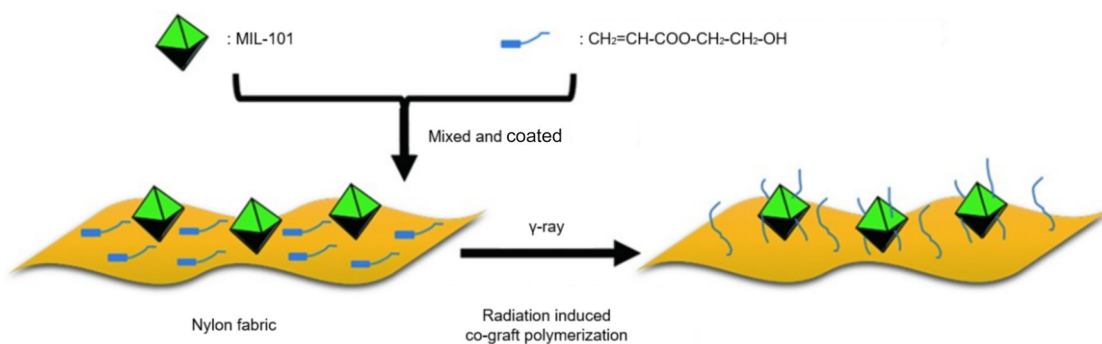


图3 辐射诱导接枝聚合制备 MOF 固定化尼龙织物的机理^[56]

Fig.3 Mechanism of the preparation of nylon fabrics immobilized with MOFs by radiation-induced graft polymerization^[56]

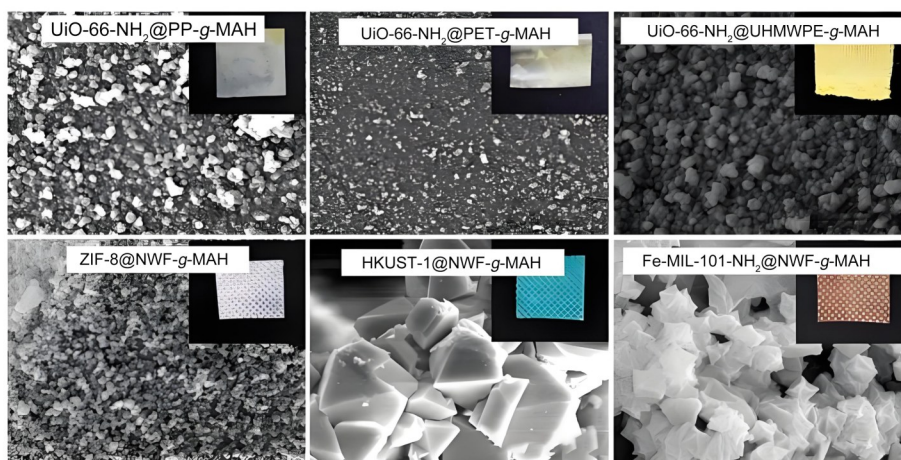


图4 间接法合成 MOF 基杂化膜

Fig.4 Synthesis of MOF-based hybrid membranes by indirect method

辐射接枝的官能团具有多样化的特点，并不局限于羧基基团^[62-64]。Wu 等^[65]通过紫外辐射诱导在聚醚砜的多孔壳侧表面接枝氨基，然后，利用原位流体处理以形成 ZIF 层，成功在柔性 PESHFs 上制备了由表面氨基诱导的高(2 2 2)取向 NH₂-ZIF-8@HF 膜。表面的氨基在底物和 ZIF 晶体

之间提供了一个键桥，促进了 ZIF 晶体的均匀成核。该策略有效地缓解了晶间缺陷，形成了高度集成和(2 2 2)定向的 ZIF-8 层。在 0.2 MPa 和室温下，NH₂-ZIF-8@HF 膜对 H₂/CO₂、H₂/N₂ 和 H₂/CH₄ 气体对均表现出优秀的理想选择性。

3 辐射诱导有机-无机杂化材料的性能研究

相较于溶胶-凝胶、共沉淀、溶剂热、化学处理等其他杂化材料制备技术,辐射诱导法(如电子束辐射、 γ 射线辐照、高能电子轰击等)具有独特的优势,它能够在分子尺度和纳米尺度上对材料结构进行精密的原位调控和功能化修饰,从而显著提升杂化材料在多个关键维度的综合性能,使得辐射诱导法制备的杂化材料在催化^[30,39,43]、分离^[58,60,66]、传感^[67-69]及航空航天^[70]、生物医疗^[27,71]等重要领域展现出广阔的应用前景。以下以辐射诱导法改善有机-无机杂化材料的催化性能、分离性能、电化学性能和机械性能为例进行介绍。

3.1 催化性能

Chen等^[39]利用高剂量电子束辐射在ZIF-8晶体的表面上形成分级孔结构和结晶ZnO纳米颗粒,制备了ZIF-8@ZnO催化剂。分级孔结构赋予了ZIF-8富集能力,具有独特的异质结构和高光催化活性的半导体结构的结晶ZnO纳米颗粒为催化限制效应提供了合适的环境。与传统水热法制备的ZIF-8@ZnO等材料相比,通过高剂量电子束制备的ZIF-8@ZnO催化剂展现出对亚甲基蓝(MB)的快速降解动力学。该材料不仅能够显著提升了印染/造纸废水中顽固染料及抗生素的处理效率,通过组分调控(如引入TiO₂、Fe₂O₃)还可进一步应用于农药分解与Cr(VI)等重金属离子的高效还原。同时,窄带隙ZnO与MOF之间的协同效应可显著促进光生电荷分离,为开发高效的光催化材料(如光解水制氢和CO₂还原催化剂)提供了新的设计思路。电子束辐射法具有优异的普适性,可用于多种MOF基杂化材料的可控合成(如HKUST-1、ZIF-67、UIO-66、MIL-101等),为进一步构筑在苛刻条件下(如高温、强酸、强碱或辐照环境)稳定高效的多功能催化剂提供了有效途径。

Zhang等^[43]通过辐射诱导法构建了HKUST-1@Cu₂O分级多孔结构。HKUST-1的多孔结构可富集反应物并提供电子转移通道,Cu₂O具有强吸附作用,二者结合实现了对硝基芳烃类污染物(如农药、染料中间体)的高效降解。其超快的反应速率、高活性和广谱性等优势使该材料在废水处理、药物合成(氨基酚制备)等领域中具有重要应用前景。不仅如此,HKUST-1@Cu₂O异质结构的电子协同

效应还可进一步拓展多功能应用,包括用于高灵敏度毒性气体检测传感器、基于高效电荷分离机制的光电器件(如太阳能电池)以及CO₂还原/析氢催化剂的设计。

3.2 分离性能

Gu等^[53]利用 γ 射线诱导接枝的方法将水溶性的PVA接枝到多壁碳纳米管(MWNT),再将接枝的g-MWNTs分散体沉积在醋酸纤维素(CA)微孔膜上,成功构建了基于多壁碳纳米的杂化膜(GMC膜)。PVA良好的亲水性使得g-MWNTs可以更稳定地分散到水中而不发生聚集,也使得g-MWNTs可以更加均匀地分散到基材表面。GMC膜表现出优异的水下超疏油性能,在十二烷、食用混合油和发动机油的油水混合溶液中,其水下油接触角(OCA)分别高达~157°、~155°和~157°。高抗油污性能和高分离性能使得GMC膜能够实现16.6 L/(m²·h·kPa)的高通量和超低压(8.4 kPa)下大于99.1%的排油率,在水包油乳液分离性能方面,优于许多先前报道的碳纳米管的复合膜。

Fu等^[72]采用高能电子轰击技术,同步实现了微晶纤维素(MCC)与甲基丙烯酸缩水甘油酯(GMA)的接枝共聚以及无机纳米粒子(纳米SiO₂、TiO₂、Fe₂O₃和Fe₃O₄)的包埋。该杂化材料对三氯乙酸(TCA)展现出优异的吸附性能:一方面,材料表面的正电荷与TCA分子的负电荷官能团产生静电引力,驱动吸附发生;另一方面,包埋的纳米粒子提供了丰富的微观孔隙和高比表面积,不仅增加了吸附位点,也促进了TCA分子的扩散与吸附。相比传统方法(如活性炭吸附、高级氧化过程、生物降解等)普遍存在的去除效率不足、操作条件苛刻等问题,这种新型杂化材料实现了高效、绿色的TCA去除(去除率高达83.27%)。鉴于饮用水氯消毒过程中,氯与有机物反应不可避免地产生消毒副产物(DBP),其中致癌风险较高的TCA含量通常最高,该材料在保障饮用水安全方面具有重要的实际应用价值。

3.3 电化学性能

Abdel Maksoud等^[67]通过辐照法将Ag纳米颗粒沉积到CaTiO₃表面,并将其均匀负载到PVA基质中,得到了PVA/Ag/CaTiO₃纳米复合膜。相较于传统的溶胶-凝胶、共沉淀、溶剂热等方法,辐照法负载的Ag/CaTiO₃ NPs具有更高的稳定性和分散

性，为载流子提供了更多的通路，从而使得该复合材料具有更加优异的光学、电学、光催化性能和更高的稳定性。并且，PVA/Ag/CaTiO₃具有成膜能力好、抗拉强度高、柔韧性强、其易加工和化学稳定性好的优点，可被广泛用于光伏器件的封装、传感器制造、降噪的电子涂层制备等方面。

Zhang等^[68]通过辐射诱导接枝再进行原位生长的方式，在PET织物上附着Fe₃O₄和Ni的纳米粒子，开发了一种高性能可穿戴柔性纺织品应变传感器(Ni@PET-g-PAO/Fe₃O₄)。Fe₃O₄纳米粒子具有高热阻和低热导率，可以抑制材料的热变形，使其表现出更好的信号稳定性。其次，Fe₃O₄纳米粒子作为PET织物和Ni沉积层之间的多功能界面层，使得Ni@PET-g-PAO/Fe₃O₄即使在500次重复拉伸后也能表现出优异的耐久性和导电性稳定性。此外，Fe₃O₄纳米粒子的引入还增强了传感器的磁响应特性，因而该传感器可用作出色的可拉伸导体和磁响应开关，在基于软质纺织品的电子器件柔性应变传感器中具有广阔应用前景。

3.4 机械性能

通过辐射诱导材料产生缺陷或发生偶联，还可以在整体上改变材料的机械性能或耐腐蚀性能。如Park等^[70]利用 γ 射线辐照产生缺陷的方法来改性碳纳米管(CNT)纱线，随后用所得的CNT纱线与双马来酰亚胺(BMI)树脂压制成复合层压板。相较于传统化学处理技术，辐射诱导交联可以穿透密集堆积的CNT纱线材料，从而实现对材料的整体改性。对CNT纱线表面的成功改性导致了含氧基团的增加，改善了纱线和树脂基体之间的润湿性和界面结合。此外，经辐射处理的CNT纱线制成的复合层压板的比拉伸模量和强度均有所提高，分别达到了258 GPa/(g·cm³)和1.89 GPa/(g·cm³)。由于具有较高的机械和电气性能，辐射法所制备的高强度CNT纱线制备复合层压板可用作高性能复合材料的主要增强材料，在航空航天领域具有很强的应用前景。

4 结论与展望

有机-无机杂化材料的制备手段多种多样，辐射诱导技术凭借其独特的能量传递形式，不论是在直接制备时的掺杂，还是间接制备时的接枝改性，都具有其他方法难以替代的优势，并且辐射效应赋予有机-无机杂化材料独特的结构也使其具

有更优异的性能。目前，辐射技术在杂化材料制备方面的机理研究仍需要深入研究，以实现辐射对杂化材料的可控性诱导。对于合成系统，更加简化、可控化的辐射合成方法仍有待开发。对于应用层面，辐射诱导法为杂化材料的合成带来了更多的组合可能性，仍有待探寻。相信在未来，随着机理研究的不断深入和材料设计的多元化，辐射诱导技术将推动更多功能材料的发展和应用。

作者贡献声明 张依帆提供资金和论文的审阅与修订。郑红星负责撰写指导和论文修订。邓鹏颺负责研究课题的指导、构思框架的提出。郭正纲、田坤遥负责资料收集和论文撰写，两位作者对工作的贡献是平等的，视为共同第一作者。黄一凡、王兴兴、刘诗涵、崔帅川负责资料收集和撰写指导。所有作者均已阅读并同意最终文本。

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