

炭黑纳米材料在电化学传感检测分析中的应用研究

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摘 要

炭黑纳米材料作为一种零维纳米结构碳材料, 其基本结构由多层级石墨微晶有序堆积构成。该材料因其大比表面积和介孔结构具有良好的吸附性能, 而且其形成的高导电碳网络结构具有优异的导电性能, 能够显著增强电化学反应过程中的电荷输运能力。在本工作中, 阐述了炭黑纳米材料增强电化学传感检测性能的基本原理, 并详细介绍了多种类型的炭黑纳米材料(VXC-72R, Super P Li, 科琴黑、乙炔黑等)及其电化学传感检测分析应用研究现状。此外, 将炭黑纳米材料与其他功能材料(碳纳米管、石墨烯、纳米黏土、MOFs等)进行复合, 可以充分发挥各组分的协同增效作用, 实现电化学传感检测性能的显著提升。本文综述了炭黑纳米材料基复合材料在电化学传感检测分析中的应用研究现状。最后, 对开发低成本、高灵敏的炭黑纳米材料基复合物传感器进行了展望。

关键词

电化学传感器, 炭黑纳米材料, 纳米复合物, 传感增强机制

Research on the Application of Carbon Black Nanomaterials in Electrochemical Sensing Detection and Analysis

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Abstract

Carbon black nanomaterials have the basic structure composed of multiple layers of ordered graphite microcrystals. These materials have good adsorption performance due to its large specific surface area and mesoporous structure, and the highly conductive carbon network structure has excellent conductivity, which can significantly enhance the charge transport ability of electrochemical reactions. In this work, the basic principle of enhancing the electrochemical sensing detection performance of carbon black nanomaterials was elucidated, and various types of carbon black nanomaterials (VXC-72R, Super P Li, Keqin black, acetylene black) and their research progress in electrochemical sensing detection applications were introduced in detail. In addition, the composites of carbon black nanomaterials with other functional materials (carbon nanotubes, graphene, nanoclay, MOFs, etc.) can fully leverage the synergistic effect of each component and achieve significant improvement in electrochemical sensing detection performance. This article reviews the current research progress of the applications of carbon black nanomaterials-based nanocomposite materials in electrochemical sensing detection and analysis. Finally, the prospect was made for the development of low-cost and highly sensitive carbon black nanomaterials-based nanocomposite sensors.

Keywords

Electrochemical Sensors, Carbon Black Nanomaterials, Nanocomposites, Enhancement Mechanisms of Sensing Detection

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1. 引言

炭黑纳米材料是一种由碳元素组成的颗粒状材料，其基本结构由多个纳米尺度的石墨片层组成，这些石墨片层形成了一种具有大表面积和多孔结构的颗粒体，使其具备了良好的吸附性能[1]-[5]。此外，炭黑纳米材料具有优异的导电性能，其石墨片层的导电性使得炭黑纳米颗粒能够形成高导电网络结构，从而在传感器、电池、催化等领域发挥着重要的作用[6]-[10]。特别是，炭黑纳米颗粒在电化学传感检测分析领域具有诸多优势。首先，该材料的大比表面积和多孔结构能够增强传感电极对被检测物的吸附能力，从而提高传感器的灵敏度[11]-[17]。其次，良好的导电性能有利于实现电荷的高效输运，增强电化学反应的速率，进而提升电化学传感器的响应速度[18]-[21]。此外，炭黑纳米材料还具有成本低、资源丰富、易于修饰等优势，这为其在电化学传感检测分析领域的广泛应用提供了有力保障。

为了增强电化学传感器的灵敏度，将炭黑纳米材料与其他纳米功能材料复合，制备功能化集成的纳米复合物增敏剂已成为传感检测分析领域的一个重要研究方向。根据公开报道的研究成果[6][7][11][13][15][22]-[26]，将炭黑纳米材料与其他功能性材料(碳纳米管、石墨烯、纳米黏土、MOFs等)进行复合，可以充分发挥各组分的协同增效作用，实现电化学传感检测性能的显著提升，如图1所示。炭黑纳米材料与碳纳米管复合，可以充分利用远程导电和近程导电的协同优势，显著增强传感器对特定物质的检测灵敏度[11][22][23]。炭黑纳米材料与石墨烯复合可以进一步显著改善纳米复合材料的导电性和机械性能，

进而提高传感器的稳定性和重复性[24][25]。此外, 炭黑纳米材料与纳米黏土、MOFs 等制备的纳米复合材料在电化学传感器中也得到了广泛的应用, 展现出了良好的应用前景[6][13][26]。

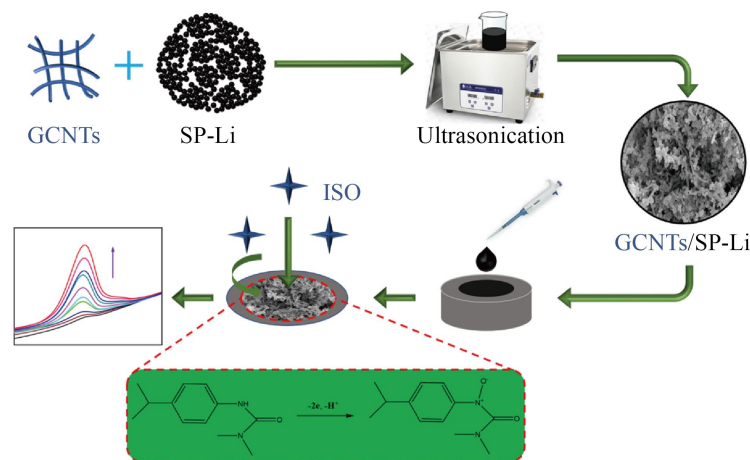


Figure 1. Synthesis procedure of the GCNTs@SP-Li/GCE sensor and the corresponding oxidation mechanism of ISO at the proposed sensor [23]

图 1. GCNTs@SP-Li/GCE 传感电极组装及其 ISO 传感检测示意图[23]

2. 炭黑纳米材料的电化学传感检测分析机理

炭黑纳米材料因其独特的结构和性能在电化学传感检测分析中发挥着重要的作用, 其传感检测分析机理主要涉及以下三个方面。首先, 炭黑纳米材料的基本结构由多个纳米尺度的石墨片层组成, 形成的珍珠链状结构具有大比表面积和多孔结构, 这能够在传感电极表面为被检测物质提供丰富的吸附位点, 有助于提高传感电极表面对被检测物的富集能力[18]-[21][27]。其次, 炭黑纳米材料因其独特的珍珠链状结构能够形成三维互联的高导电碳网络结构, 能够有效地促进电荷传输, 降低传感电极与电解液之间的界面电阻, 提高传感电极的导电性和电催化活性, 有助于增强传感器对目标检测物的检测灵敏度[28]-[32]。此外, 炭黑纳米材料还具有一些独特的表面性质和化学活性, 可在电化学传感检测的过程中与被检测物相互作用, 实现对目标物质的选择性检测[18][33]-[37]。需要注意的是, 炭黑纳米材料还可以与其他纳米材料复合形成异质结结构, 产生协同增效效应, 这能够进一步增强其电化学传感检测分析性能[23][26][38][39]。

3. 炭黑纳米材料类型

炭黑纳米材料主要涉及 VXC-72R、Super P Li、科琴黑、乙炔黑等类型。这几种材料在微观结构、导电性能以及传感应用方面有所不同。VXC-72R 因其高比表面积和一定量的羧基含量, 可在高灵敏电化学传感检测应用中发挥重要的作用[7][40]。Super P Li 因其超低灰分特性能够保障生物分子检测特异性, 在葡萄糖传感检测中展现较宽的线性检测范围。科琴黑具有超高比表面和一定的介孔体积, 可显著缩短传感检测响应时间, 但机械强度低的特点会限制该材料的结构稳定性[15][34]。乙炔黑的导电性相对较差, 但是该材料的成本低, 有助于实现传感器件的规模化生产应用[35][36][41][42]。

3.1. VXC-72R 炭黑纳米材料

VXC-72R 炭黑纳米材料是一种高品质的导电炭黑, 具有高导电性、低硫含量和低灰分含量等特性。该材料的物理形态为粉状, 比表面积较大, 粒径较小, 着色力强, 密度较低, 使其在多个领域都有广泛

应用[40] [43]-[45]。在电化学传感检测分析中, VXC-72R 炭黑纳米材料可提高传感器的导电性能和吸附能力, 增强对目标物质的检测灵敏度。Zhao 等人采用简单有效的超声波辅助策略制备了 VXC-72R 纳米颗粒材料修饰铂基金属有机骨架传感器, 如图 2 所示[7]。VXC-72R 纳米粒子具有良好的导电性能和良好的分散性, 可以导电形成相互连接的碳导电通道。此外, VXC-72R 纳米粒子的引入可以有效地弥补铂基金属有机骨架的导电性能。所构建的电化学传感器具有良好的 MP 检测性能, 检测限可达到 1.35 nM (线性 MP 浓度范围: 0.01~10 μ M)。当用于检测果汁样本中的 MP 时, 该传感器显示可接受的回收率为 96.00%~104.67 %。

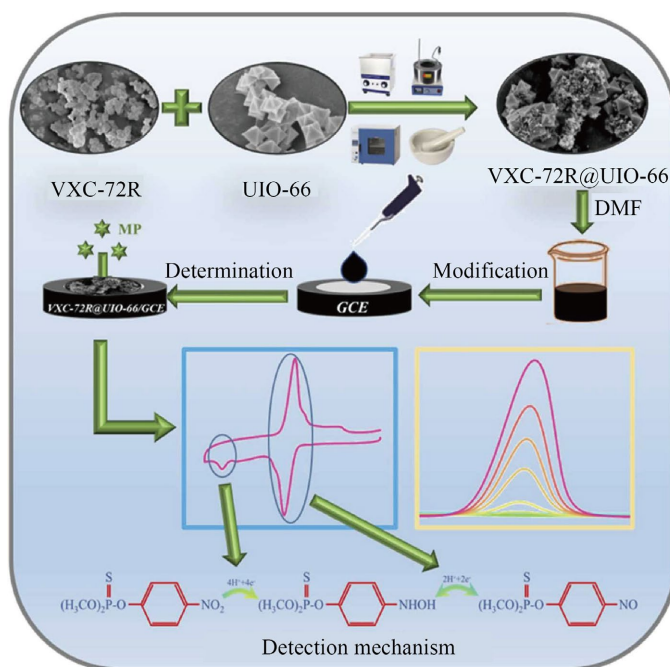


Figure 2. Fabrication of VXC-72R@UIO-66/GCE sensor for the MP detection [7]

图 2. VXC-72R@UIO-66/GCE 传感器组装及其 MP 检测示意图[7]

3.2. Super P Li 炭黑纳米材料

Super P Li 炭黑纳米材料是一种高性能的导电炭黑, 具有较小的纳米颗粒粒径和较高的比表面积, 使其在电化学传感检测中具有独特的优势。该材料优异的导电性能可以有效地提高传感电极的电导率, 降低电极与电解液之间的接触电阻, 从而增强传感电极表面电化学反应的效率[6] [26] [43]。近年来, Super P Li 炭黑纳米材料在电化学传感器中的应用也受到了越来越多的关注。Liu 等人使用 β -环糊精功能化的 Super-P-Li 炭黑纳米材料修饰玻碳电极, 构建了一种新型 MP 检测用电化学传感器[18]。具有高电化学活性面积的 SPLCB 表现出优异的导电性, 这有利于形成互连的高导电碳网络结构。 β -CD 功能化不仅可以促进 SPLCB 的均匀分散, 还可以进一步增强传感电极表面对 MP 分子的富集能力。此外, SPLCB 的存在可以弥补 β -CD 电导率低的缺陷。在最佳实验条件下, SPLCB 和 β -CD 的协同增效作用实现了传感器的高灵敏 MP 检测性能。此外, 该传感器还成功用于苹果汁中的痕量 MP 的检测分析。

3.3. 科琴黑纳米材料

科琴黑纳米颗粒是一种具有特殊结构的炭黑材料, 因其独特的支链状和多晶体形态能够形成高效的

高导电碳网络结构。该材料较少的添加量就可以显著提升导电率,有效降低传感电极与电解液的界面内阻,进而提高电化学反应过程中电极表面的电荷传输效率[8][14][28][34]。Zhang 等人通过超声波分散科琴黑纳米材料,然后涂覆在改性碳布电极上,获得了一种简单低成本的电化学传感器,实现了重金属离子 Cr(VI)的高灵敏检测[14]。研究结果表明,修饰传感电极的电荷输运能力得到显著增强,这主要得益于科琴黑优异的导电性能。此外,科琴黑纳米材料的大比表面积和介孔特征能够增强传感电极表面对被检测物的富集能力。在最佳实验条件下,电化学传感检测 Cr(VI)的还原峰电流与其在样品中的浓度之间的关系。还原峰电流强度与其浓度在 0.025~57 μM 和 57~483 μM 范围内呈良好的线性关系。该传感器的检出限可达到 9.88 nM ($S/N = 3$),显示出优异的重金属离子检测性能。

3.4. 乙炔黑纳米材料

乙炔黑炭黑纳米材料是一种由乙炔热解生成的高性能导电炭黑,具有高纯度、低杂质含量以及优良的导电性和吸液性[17][30][35]。其粒径小、比表面积大,表观密度低,呈现多孔结构,这使其具备良好的吸附性能。在导电应用中,乙炔黑炭黑纳米颗粒能有效提升材料的导电性能,有助于提高电化学传感器的灵敏度[5][20][24][25]。Sun 等人基于乙炔黑修饰的玻碳电极开发了一种阿霉素检测用电化学传感器[30]。在最佳条件下,所开发的传感器表现出令人满意的选择性、可重复性和稳定性。此外,根据阿霉素的电化学传感检测结果,随着阿霉素浓度的增大,阿霉素的电化学传感响应值也不断增大,而且二者具有较好的线性拟合关系,检测限可达到 3.006 nM。此外,将开发的传感器用于人血清样品中阿霉素的定量分析,表现出令人满意的回收率。

4. 炭黑纳米材料基复合物在电化学传感检测分析中的应用

利用固相法制备高功率尖晶石型 LiMn_2O_4 正极材料时,需要选择合适的固相反应策略。同时还需要注意的是,不同的锰源(如 MnO_2 、 MnCO_3 、 Mn_3O_4 、 MnO 等)具有不同的结构和形貌,将其作为固相反应物制备的尖晶石型 LiMn_2O_4 正极材料也会表现出不同的电化学性能[25][31][38][42]-[45]。因此,在制备高功率尖晶石型 LiMn_2O_4 正极材料时,也要为固相反应选择合适的锰源。

4.1. 炭黑纳米材料@碳纳米管复合材料

炭黑纳米颗粒@碳纳米管复合物能够结合炭黑纳米材料和碳纳米管的优良特性,二者结合形成协同效应,优势互补[11][22][23]。一维中空结构的碳纳米管和炭黑纳米颗粒的复合能够发挥远程导电和近程导电的协同增效作用,可进一步增强炭黑纳米材料的导电性。此外,炭黑纳米材料有助于改善碳纳米管易团聚等问题,使复合材料性能优于单一组分,在电化学传感应用表现出更好的检测分析性能。Li 等人通过简单的超声波辅助策略制备了 SuperP Li 碳纳米颗粒和石墨化多壁碳的纳米复合材料(GCNTs@SP-Li),并用于修饰玻碳电极构建 GCNTs@SP-Li 纳米复合物传感器,实现异丙隆(ISO)的高灵敏检测[23]。对于 GCNTs@SP-Li 纳米复合材料、多孔结构的 GCNT 具有优异的吸附性能,这有助于 ISO 的预富集并促进电解质渗透。此外,GCNT 因其较高的石墨化程度而具有良好的导电性。GCNTs@SP-Li 纳米复合材料法互连线状结构显著缩短了电子传输距离,从而显著加速了氧化还原反应的过程。GCNTs 和 SP-Li 的多功能集成实现了 GCNTs@SP-Li/GCE 传感器对 ISO 的灵敏检测。此外,该传感器在测定河水和自来水中的 ISO 时表现出令人满意的回收率。

4.2. 炭黑纳米材料@石墨烯复合材料

石墨烯具有优异的导电性、高比表面积和良好的机械性能,与炭黑纳米颗粒复合后,可以进一步提高复合材料的导电性能和吸附性能。在电化学传感检测领域,炭黑纳米颗粒@石墨烯复合材料被广泛应

用于检测生物分子、环境污染物和食品中的有害物质等[24] [25]。Deng 等人使用乙炔黑和石墨烯的纳米复合材料修饰碳糊电极, 构建了一种色氨酸(Trp)检测用电化学传感器[24]。由于高吸附能力、大表面积和众多活性位点, 该复合材料对 Trp 的氧化显示出很明显的增强作用, 并显著提高了传感检测 Trp 的峰值电流。在 $1.0 \text{ M H}_2\text{SO}_4$ 中, 色氨酸和酪氨酸(Tyr)的伏安响应很好地分为两个不同的峰, 峰电位差(ΔE_{pa})为 115 mV 。在优化条件下, 传感检测色氨酸的氧化峰电流与其浓度在 $0.1 \mu\text{M}$ 至 0.1 mM 的范围内成正比, 检测限为 60 nM ($S/N = 3$)。

4.3. 炭黑纳米材料@纳米黏土复合材料

炭黑纳米材料@纳米黏土复合材料在电化学传感检测中具有显著优势。炭黑导电材料的导电性可降低电极界面电阻, 加速电子传输, 而纳米黏土能够提供丰富的活性位点, 二者协同的优势有助于增强电化学信号响应[26] [40]。对于该类型的复合材料, 调节炭黑含量可调控复合材料的导电性能, 满足不同传感器需求, 实现对目标物质的高灵敏检测。此外, 炭黑和纳米黏土制备成本低、原料来源广泛, 有利于降低电化学传感器的生产制造成本。Li 等人提出了一种简单、低成本、可扩展的超声波辅助策略, 用于制备 Super-P 炭黑纳米颗粒分散的凹凸棒石(Pal)纳米纤维复合材料, 如图 3 所示[26]。该复合材料进一步修饰玻碳电极, 构建 DQ 检测用的电化学传感器(Pal/Super-P/GCE)。该传感器在 $0.00050 \sim 1.0 \mu\text{M}$ 的线性 DQ 浓度范围内表现出超灵敏的传感检测性能, 检测限可达到 0.1514 nM 。当应用于检测河水、马铃薯和黄瓜中的 DQ 时, 所构建的传感器的回收率范围为 95.6% 至 100.3% 。如此优异的 DQ 检测性能主要是由于 Pal 和 Super-P 的协同作用。Pal 具有高吸附能力和优异的生物相容性, 由于其优越的离子交换性能和表面丰富的 Si-OH, 可以为 DQ 吸附提供许多附着位点。具有珍珠链状结构的 Super-P 有助于形成相互连接的导电网络, 从而促进 Pal 的均匀分散, 并弥补 Pal 导电性差的问题。

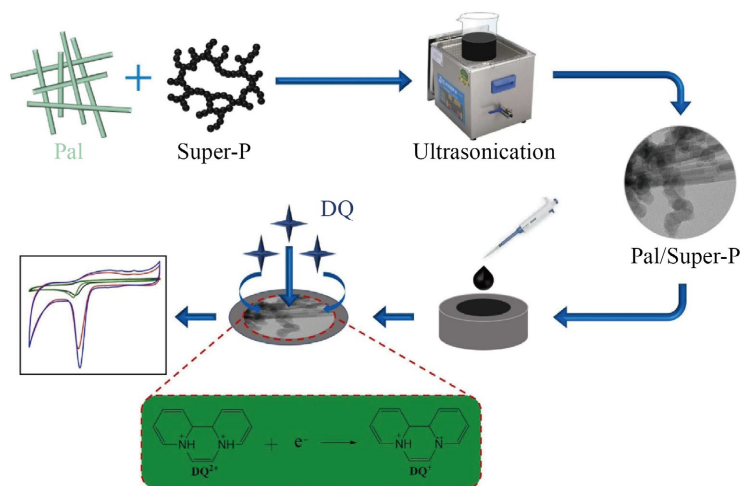


Figure 3. Fabrication of the Pal/Super-P nanocomposite and its application for the electrochemical detection of DQ [26]

图 3. Pal/Super-P 纳米复合物修饰的传感器应用于 DQ 电化学检测示意图[26]

4.4. 炭黑纳米材料@MOFs 复合材料

炭黑纳米材料@MOFs 复合材料是一种将炭黑纳米材料与金属有机框架材料(MOFs)复合而成的新型复合材料。炭黑纳米颗粒的加入可以改善 MOFs 的导电性能和稳定性。在电化学传感检测领域, 炭黑纳米颗粒@MOFs 复合材料能够展现出独特的优势[6] [13] [15] [22]。Jin 等人通过水热法合成了一种高催化

活性的导电铜基金属有机框架($\text{Cu}_3(\text{HHTP})_2$), 并使用炭黑 Super-P 进一步修饰 $\text{Cu}_3(\text{HHTP})_2$ 获得功能化集成的纳米复合材料[6]。将该复合材料用来修饰玻碳电极, 构建了一种甲基乙二醛(MG)检测用电化学传感器(Super-P/ $\text{Cu}_3(\text{HHTP})_2/\text{GCE}$)。由于 $\text{Cu}_3(\text{HHTP})_2$ 的催化活性和 Super-P 的增强导电性, 所构建的传感器具有较宽的线性检测范围($1\sim 500\ \mu\text{M}$)和低检测限($0.0275\ \mu\text{M}$)。此外, 该传感器已成功应用于牛奶、啤酒、尿液和人血清样本中 MG 的测定, 回收率范围为 91.7%至 115.7%。

5. 结论

炭黑纳米材料凭借其独特的结构和性能, 在电化学传感检测领域展现出巨大的应用潜力。本研究探讨了炭黑纳米材料增强电化学传感检测性能的基本原理, 揭示了其在电化学传感器中的关键作用机制。通过介绍多种类型炭黑纳米材料(VXC-72R、Super P Li、科琴黑、乙炔黑等)的研究现状, 可充分了解不同种类的炭黑纳米材料在电化学传感检测分析中的应用优势。此外, 本文还详细综述了炭黑纳米材料与碳纳米管、石墨烯、纳米黏土、MOFs 等形成的复合材料在电化学传感检测分析中的研究现状。这些复合材料结合了炭黑纳米材料与其他材料的优良特性, 实现了性能的协同增效。尽管炭黑纳米材料及其复合材料在电化学传感检测领域已经取得了显著的研究成果, 但仍存在一些挑战和改进空间。未来, 还需要进一步深入研究复合材料结构与性能之间的关系, 优化复合材料的制备工艺, 提高复合材料的稳定性, 实现炭黑纳米材料及其复合材料的大规模、低成本生产。同时, 应加强跨学科合作, 结合材料科学、化学、生物学、物理学等多领域的知识和技术, 推动炭黑纳米材料基复合物传感器的创新和发展, 为其在环境监测、生物医学、食品安全、工业控制等领域的广泛应用奠定坚实基础。总之, 随着研究的不断深入和技术的不断进步, 炭黑纳米材料基复合物传感器有望在未来的电化学传感检测领域发挥更加重要的作用, 为解决实际检测问题提供更加有效和可靠的解决方案。

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