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Application of polydopamine coating in polyetheretherketone modification

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Abstract: *Poly-ether-ether-ketone (PEEK), a new type of bone implant, as a substitute for titanium and other traditional bone implants, its clinical application is affected by its surface hydrophobicity and biological inertia. The research on the improvement of osteogenesis and antibacterial activity of PEEK has attracted more and more attention. Polydopamine (PDA) coating, a widely used surface modification coating, is used to modify PEEK, which can not only solve the problem of low surface activity of PEEK, but also maintain the good mechanical properties of PEEK. In this paper, the articles of surface modification of PEEK with polydopamine coating are summarized and reviewed.*

Keywords: *Polyetheretherketone, Polydopamine, Surface modification*

1. Introduction

Polydopamine can adhere to almost any substrate material, which can increase the activity of the material surface and provide a secondary modification platform for grafting substances, thus it has been widely used in the surface modification of bone implant materials since its discovery. Meanwhile, PDA is biocompatible and is a natural melanin that does not trigger an immune response[1].

However, PDA solution oxidation also has some limitations. For example, the molecular mechanism involved in the polymerization of PDA has not been fully clarified. Many key factors in the process of solution oxidation will significantly affect the deposition speed and thickness of PDA, such as reaction time, monomer concentration, PH and temperature. However, PDA provides excellent adhesion to many materials and even superhydrophobic surfaces with controllable and stable deposition results. Based on its strong adhesion and excellent biocompatibility, DA is expected to be used to improve the surface adhesion of materials and promote cell adhesion, thus preparing ideal bone repair materials.

Polyetheretherketone has excellent properties such as corrosion resistance, high temperature resistance, and good biocompatibility. The modulus of elasticity of PEEK is 4. 1 Gpa, which is closer to that of human bone tissue than titanium[2][3]. PEEK has been widely used in medical fields such as cranial reconstruction, intervertebral fusion, oral implants[4]. However, polyetheretherketone is highly biologically inert, which is not conducive to osseointegration of implants, and has poor antimicrobial properties. How to increase the bioactivity of PEEK to better apply it in medical implants and other fields has received increasing attention from scholars.

Some researchers have incorporated bioactive materials such as hydroxyapatite into PEEK to achieve a co-blended modification that improves the bioactivity of PEEK[5]. In contrast to this approach, surface modification is simpler and does not alter the mechanical properties of polyetheretherketone. The construction of polydopamine coatings on the surface of PEEK is a simple and economical modification strategy. By impregnation, not only can the hydrophilicity of the material surface be improved, but also a large number of bioactive substances and drugs can be deposited on the PEEK surface to improve the osteogenic and antimicrobial properties of PEEK.

2. Intruduction of polydopamine and its reaction mechanism

Marine mussels have strong adhesion ability and can be fixed on the surface of various substrates including polytetrafluoroethylene. Studies have found that the mussels'adhesive proteins secreted by it

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contains a certain amount of L-3, 4-dihydroxyphenylalanine (DOPA) and a small amount of lysine residues. The substance achieves firm adhesion through coordination cross-linking and covalent and non-covalent interactions of ortho-diphenol groups in DOPA[6][7]. Dopamine Dopamine (DA), a derivative of DOPA, has an adhesion capacity similar to that of dopa. Messersmith et al. [8]first reported that under aerobic conditions, in Tris buffer at pH=8. 5, the small biological molecule DA can oxidize and self-polymerize to form PDA, which can be deposited by simple impregnation on almost any material to form thin and strong PDA films with thicknesses ranging from a few nanometers to 4100 nm.

At present, the adhesion mechanism of polydopamine has not been fully elucidated, but most scholars believe that dopamine is oxidized to polyquinone[9], amino cyclization followed by oxidative rearrangement to form 5, 6-dihydroxyindole, and finally self-polymerization occurs[10]. Use of polydopamine coating can significantly improve cell adhesion[11], increase the hydrophilicity of the material surface, and can also be used as an intermediate layer to graft drugs, growth factors modified bone implants.

3. Application of polydopamine in PEEK modification

3.1. Improving the osteogenic properties of PEEK

As an inert material, PEEK is somewhat limited in clinical applications due to its poor osteogenic activity. PDA coatings can provide a platform for grafting reactions to functionalize the PEEK surface and successfully immobilize proteins, peptides, metal ions, and other substances with osteogenic inducing effects onto the PEEK surface.

3.1.1. Fixation of bioactive substance

Modification of the surface of bone implants with osteogenic-related active proteins and peptides can significantly improve osseointegration. The large number of catechol groups and amino groups contained in DA is capable of immobilizing osteogenic active substances through covalent and non-covalent linkages.

Bone morphogenetic protein 2(BMP-2) promotes the targeted differentiation of bone marrow mesenchymal cells into osteoblasts[12]. The strategy of anchoring BMP-2 to the implant surface via PDA promotes bone tissue regeneration[13][14]. However, during the reaction, some BMP-2 inevitably loses its activity. Qin et al. [15]encapsulated PLGA microspheres to achieve continuous release of BMP-2 nanoparticles, and fixed the microspheres by adhering to the PEEK surface through PDA. In the composite, pBMP-2 release was controlled and the BMP-2 protein was produced continuously and efficiently after transfection, promoting the recruitment, osteogenic induction and differentiation of stem cells on the PEEK surface and accelerating the process of bone repair after PEEK implantation. In addition to the immobilization of PLGA microspheres, chitosan/mesoporous silica nanoparticles (CS/MSNs) composite microspheres were also successfully immobilized on the surface of the sulfonated PEEK scaffold, which could continuously release platelet-derived growth factor BB (PDGF-BB) and the small organic compound kartogenin, which enhance cartilage differentiation of BMSCs in vitro and promote cartilage regeneration in vivo[16]. Giwan et al. [17]showed that uniform collagen or insulin layer could be deposited on PEEK with water-soluble carbodiimide activation agent after forming PDA film on PEEK surface, and covalent fixation of collagen or insulin on PEEK surface was conducive to adhesion, proliferation and osteogenic differentiation of MC3T3-E1 cells.

For 3D printed PEEK scaffolds, the ideal coating can also be constructed by impregnation using PDA. Ma et al. [18]implanted extracellular vesicles on a 3D-printed PEEK scaffold fixed with polydopamine into New Zealand white rabbits, and showed that this scaffold showed excellent osteogenic ability after 6 and 12 weeks through in vivo imaging, microCT, histology and immunohistochemical analysis. The cell adhesion sequence of arginine-glycine-aspartate (Arg−Gly−Asp, RGD) found in fibulin can bind to integrin receptors to mediate cell adhesion[19]. PDA can integrate RGD peptide onto PEEK surface after sulfonation and oxygen plasma treatment to obtain a biomolecular layer with a density of 42. 934ng/cm^2. Surface modification of RDG peptide promoted osteogenic differentiation of cells compared to the PEEK surface, and biomineralization results in simulated body fluids confirmed the superior bioactivity of the modified PEEK in vitro[20] .

3.1.2. Fixed metal ions

Increasing the bioactivity of PEEK materials can be achieved by the use of metal ions in addition to the release of bioactive molecules. There are two broad methods for modifying PEEK using metals. The

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first method is to modify PEEK by doping the metal directly into the interior of the PEEK, which will affect the mechanical properties of the PEEK to some extent. Another method is to immobilize metal ions on the surface of PEEK. The PDA can adhere the metal ions to the PEEK surface through chelation.

Magnesium is one of the main mineral components of the bone matrix and promotes osteogenesis and angiogenesis[21]. Some studies have combined 3D printing technology, PDA coating and magnesium ion surface modification to prepare a porous PEEK scaffold with a composite magnesium coating. In vitro, the coating is beneficial to cell adhesion and proliferation, bone formation and blood vessel formation on the scaffold surface. In vivo, magnesium loading significantly improved the bone integration capacity of the PEEK scaffold, and the released magnesium ions promoted early vascular growth and thus enhanced the inward growth of the porous PEEK scaffold bone[22]. Manganese (Mn) is an essential trace element that plays a role in bone formation and bone tissue metabolism[23]. Yan et al. [24]used PDA bionic adhesive coating to fix manganese on PEEK surface. The addition of Mn increased the expression of osteogenic genes, alkalinephosphatase (ALP) and mineralization, and in vivo experiments demonstrated its ability to promote new bone growth.

3.2. Improving the antibacterial properties of PEEK

It has been shown that at 48h, the number of Staphylococcus aureus (S. aureus) and Escherichia coli (E. coli) biofilms on the PEEK surface is approximately two orders of magnitude higher than that of conventional bone implants made of titanium[25]. The poor antimicrobial performance of the implant causes a number of problems such as infection and aseptic loosening after implantation, potentially leading to implant failure. PDA can provide anti-microbial properties to implant materials through strategies such as deposition of metallic substances, drug loading, and adhesion of anti-microbial molecules.

3.2.1. Fixation of antibiotics

Sun et al. [26]constructed a polydopamine and gentamicicinsulfate (GS) layer on the surface of sulfonated porous PEEK (SPEEK). The successful loading of GS can effectively control the development of infection and inflammation, and is conducive to tissue repair. The antibacterial rate of S. aureus and E. coli reached almost 100% within 24 hours, and the antibacterial effect was effective in 3 days, with lasting bacterial clearance. SPEEK-PDA-GS has a good healing effect on infected bone defects and promotes the formation of new bone. However, constructing an antibiotic slow-release system on the PEEK surface can effectively enhance the antimicrobial effect. One study prepared vancomycin gelatin nanoparticles (Van GNPs) using a two-step desolvation method. The Van GNPs were uniformly distributed and sized on the PEEK surface, and antimicrobial experiments showed that the antibacterial effect of PEEK on S. aureus and S. mutans was significant after binding to Van GNPs[27].

3.2.2. Fixation of metal ions

Silver, a commonly used broad-spectrum antimicrobial agent, has strong antimicrobial properties and is not susceptible to drug resistance. PDA can chelate silver ammonia ions to reduce them to silver nanoparticles (AgNPs) and then immobilize them on the surface of PEEK[28].

AgNPs can interact with cell structure and biomolecules while generating high levels of reactive oxygen species (ROS) by anchoring to the bacterial cell wall and can bind to proteins present in the cell membrane when penetrating inside the microbial cell. As a result of increased ROS levels, cells undergo apoptosis-like reactions, lipid peroxidation and DNA damage[29]. Studies have shown that nanosilver coatings formed on PEEK surfaces exhibit excellent antibacterial properties in vitro and in vivo with good biocompatibility[30].

3.2.3. Immobilization of antimicrobial peptides

Antimicrobial peptides (AMPs) have a broad spectrum and powerful antimicrobial ability. Even for fungal and multi-drug resistant microorganisms, the antimicrobial peptides can still produce the desired antimicrobial effect[31]. Some studies have used the antimicrobial peptide KR-12 and PDA to modify the surface of PEEK. In vitro and in vivo antimicrobial tests showed that the modification of KR-12 resulted in good antimicrobial activity of PEEK against methicillin-sensitive Staphylococcus aureus. In addition, the modified PEEK could significantly enhance the adhesion, proliferation and differentiation of rBMSCs. In vivo experiments also show that KR-12 coating has osteogenic ability[32].

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3.3. Enhance antibacterial property and osteogenic differentiation

PDA can also be used to immobilize both antimicrobial and osteogenic substances on the PEEK surface to achieve the best of both worlds, creating a bifunctional PEEK with antimicrobial properties and enhanced osseointegration.

It has been studied that the adhesion of Mn ion and Ag ion to the surface of PEEK can obviously promote the adhesion of MC3T3-E1. Firstly, the surface of PEEK was modified by polydopamine (PDA) coating by incubating in dopamine solution. Then, PEEK-PDA was modified by manganese (Mn) and silver (Ag) ions by impregnation method, and PEEK-PDAMn/Ag was prepared. The results of osteogenic gene expression, ALP and alizarin red staining showed that PEEK-PDA-Mn and PEEKPDA-Mn/Ag had good osteogenic induction. In addition, PEEK-PDA-Mn/Ag has extensive antibacterial activity against Staphylococcus aureus and Escherichia coli in vitro[33].

Wang et al. constructed a multifunctional nanocoating consisting of GO, PDA coating, and bone-forming peptide (BFP) on PEEK implants. It is an osteoinductive oligopeptide produced by bone morphogenetic protein-7 (BMP-7), which is composed of 15 amino acids (GQGFSYPYKAVFSTQ sequence)it has better osteoinductivity than BMP-7. The loading of this oligopeptide endows PEEK with the function of osteointegration in vivo, and the combination of PDA and GO nanosheets, Produced asynergetic photothermal/photodynamic effect. PDA has a good near-infrared absorption ability, which can enhance the light-harvesting ability of GO nanosheets. It can generate high temperature and a large amount of ROS under the light irradiation of 808nm wavelength, and perform powerful antibacterial phototherapy on two-dimensional nano-coatings, while not as Resistance to antibiotics as well[34].

There have also been studies using silver and peptide binding methods to immobilize Ag to PEEK surfaces using PDA coatings. Carboxymethyl chitosan(CMC) fixed to the surface of PEEK by spinning, can control the release of silver ions while enhancing the antibacterial effect, resist the side effects caused by silver ions, and play a synergistic antibacterial effect with silver ions. At the same time, BFP was modified onto the PEEK surface by 1-(3-dimethylaminopropyl)-3-ethylcarbonimide hydrochloride (EDC)/N-hydroxy succinimide (NHS). The PEEK-Ag-CMC-BFP was successfully prepared. The experiment showed that the successful modification of the coating improved the hydrophilicity of PEEK, which was beneficial to cell adhesion and proliferation. The antibacterial ring and antibacterial curve and the observation of bacterial morphology under SEM indicated that it was under the control of CMC, the release of silver ions significantly enhanced the antibacterial ability of PEEK. BFP was added to improve the osteogenic differentiation ability of PEEK to prepare PEEK implants with dual functions[35].

Composite PEEK mixed with carbon fibers is also widely used clinically. Carbon fiber is often used as a filler to enhance the mechanical strength of PEEK. Carbon fiber is often used as a filler to enhance the mechanical strength of PEEK. Compared with pure PEEK, carbon fiber-reinforced polyetheretherke (CFR-PEEK) has better mechanical properties, can better bear the load in the load-bearing area, effectively reduces stress shielding. However, it is still a biologically inert material, and PDA can also modify surface of CFR-PEEK. Some studies have combined photosensitive antibacterial coatings, covering the surface of sulfonated porous CFR-PEEK with PDA-$Ti_3C_2T_x$ coatings. $Ti_3C_2T_x$ is a layered 2D titaniumcarbide (MXene) with an ultra-thin structure, and its antibacterial osteogenic activity increases with the concentration of nanosheets. And enhanced. The MXene-modified surface achieves the dual therapeutic capabilities of CFR-PEEK composites, including photothermal antibacterial activity and osteogenic activity, with only one photothermal material, $Ti_3C_2T_x$[36].

4. Conclusions

Surface modification of PEEK by polydopamine coating as an intermediate layer is an economical and simple method. The active functional groups provided for the surface of PEEK can further adhere to drugs, proteins and other modified substances, which can effectively improve the biological activity of PEEK. Its mediated surface modification can prepare PEEK surface with excellent antibacterial and osseointegration properties. The PDA coating enhances the osteogenic activity of PEEK by immobilizing biologically active substances and metal ions, and improves the antibacterial activity of PEEK by immobilizing metal ions, antibiotics, AMP and can also prepare dual-functional modified coatings applications have been widely used.

However, the polydopamine coating itself is prone to agglomeration during the deposition process, which can result in insufficient adhesion, poor adhesion, and non-uniform coating. To improve the deficiencies of the PDA coating, it is necessary to study further the molecular mechanism of the PDA

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deposition process and the factors that may affect the deposition of PDA on different substrate materials, such as temperature, time, pH, hydrophobicity of the substrate material, and roughness of the substrate material, in order to Improve the adhesion effect of PDA coating, and provide some theoretical guidance for polydopamine coating modification of PEEK.

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