Global Journal of Science Frontier Research: B
Chemistry
Volume 25 Issue 1 Version 1.0 Year 2025
Type: Double Blind Peer Reviewed International Research Journal
Publisher: Global Journals
Online ISSN: 2249-4626 & Print ISSN: 0975-5896

Advances in Acid and Post-Graphitization Treatments for Mesophase Pitch-based Carbon Fibers: A Review

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Abstract- The complete process of preparing pitch-based carbon fibres from mesophase pitch has been extensively studied by numerous researchers. This process mainly includes mesophase pitch preparation, melt spinning, stabilization (pre-oxidation), carbonisation, and graphitisation. While each stage has been comprehensively and deeply investigated, the treatment methods and operations that determine the effectiveness of these steps have not yet received adequate attention or analysis. This paper introduces the acid treatment process between mesophase pitch preparation and melt spinning, as well as the post-treatment procedures following the production of graphitized fibres, aiming to provide technical guidance for researchers in related fields.

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GJSFR-B Classification: LCC: TA418.76

Advances in Acid and Post-Graphitization Treatments for Mesophase Pitch-based Carbon Fibers: A Review

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I. INTRODUCTION

With the widespread application of mesophase pitch-based carbon fibres in both military and civilian sectors[1,2], China has invested significant time and funding into the development of production technologies for these fibres[3-7]. While notable progress has been made, a performance gap still exists between domestic fibres and those produced in Japan and the United States[8-10]. Comprehensive research has been conducted across the entire manufacturing process, including pitch preparation[11-13], melt spinning[14,15], pre-oxidation treatment[16,17], carbonisation[18,19], and graphitisation processes[20,21], leading to substantial technical advancements. In recent years, researchers have found that proper acid treatment after mesophase pitch preparation can significantly improve the effectiveness of melt spinning[22]. Additionally, optimized post-treatment of graphitized fibres can greatly enhance their overall performance[23]. This paper presents a summary and analysis of recent findings related to these two key yet often overlooked process stages.

II. ACID TREATMENT

In the fabrication of mesophase pitch-derived carbon fibers, acid treatment constitutes an essential pretreatment step that significantly influences the subsequent carbonization and graphitization behavior [24]. This pretreatment primarily enhances mesophase pitch spinnability, improves oxidative stabilization efficiency, and ultimately tailors the microstructure and performance of derived carbon fibers. However, in recent years, this topic has received limited dedicated research attention, resulting in a scarcity of relevant studies. This section provides a detailed analysis of the purposes, mechanisms, and process parameters involved in acid treatment.

a) *Main Objectives of Acid Treatment*

The primary purposes of acidification treatment include[25,26]:

i. *Modifying Molecular Structure*

Through treatment with acid reagents—primarily nitric acid, sulfuric acid, or mixed acids—oxygen-containing functional groups such as carboxyl, hydroxyl, and carbonyl are introduced into mesophase pitch molecules. These functional groups increase the polarity and chemical reactivity of the pitch. Controlled cross-linking induces thermosetting behavior in the pitch system, with the raised softening temperature (ΔT ~15-25°C) directly correlating with improved spin-line stability during melt extrusion.

ii. *Optimization of Melt-Spinning Capability*

Acid treatment yields superior rheological stability, suppressing spin-line fractures and diameter fluctuations by >40%. The mild cross-linking between molecular chains helps suppress deformation caused by excessive flow of pitch melt under high-temperature spinning conditions. Collectively, these optimizations promote superior spinnability in the mesophase pitch.

iii. *Enhancing Oxidation Efficiency*

The reactive sites introduced during acid treatment accelerate crosslinking reactions in the subsequent pre-oxidation process. The optimized protocol concurrently enhances oxidative stabilization

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kinetics and minimizes structural imperfections in resulting fibers.

iv. *Increasing Carbonisation Yield*

The acid-induced crosslinking networks significantly suppress volatile emission during pyrolysis (TGA-verified mass loss reduction >40%), leading to superior carbon fiber yields exceeding 80% compared to <60% for untreated counterparts.

b) *Mechanism of Acid Treatment*

During the chemical reaction process, acids—exemplified by concentrated nitric acid—react with aromatic hydrocarbons in pitch via nitration, oxidation, and sulfonation reactions, resulting in the formation of oxygen-, nitrogen-, and sulfur-containing functional groups. The underlying reaction mechanism (Fig. 1 [27]) involves thermally activated condensation between carboxyl (–COOH) and hydroxyl (–OH) functionalities, forming extensive crosslinked networks through ether (−O−) and ester (−COO−) bridged structures. The significantly enhances intermolecular interactions. The resulting crosslinked network can suppress the loss of anisotropic properties in mesophase pitch during melt spinning, thereby maintaining the liquid crystalline order.

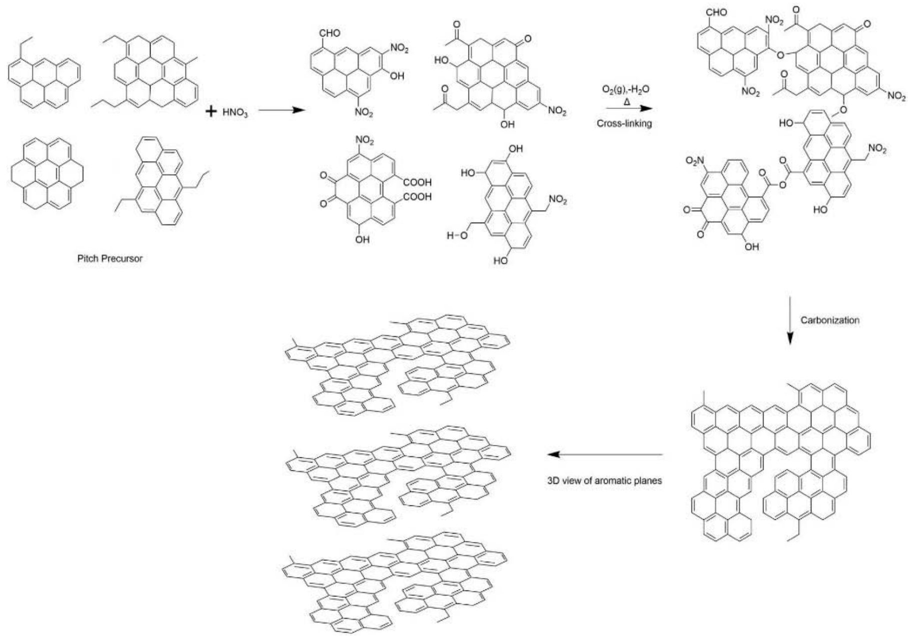

Fig. 1: The general reaction mechanism of acidification treatment

c) *Process Parameters of Acid Treatment*

The process parameters involved in acid treatment, their corresponding effects, and typical operating conditions are summarised in the table below[28-30]:

Acidification Process Parameters

Parameter	Influence	Typical Conditions
Acid Type	Nitric acid (strong oxidising property), sulfuric acid (good cross-linking effect), mixed acid (synergistic effect)	65%-98% HNO_3 or H_2SO_4/HNO_3 mixture
Acid Concentration	Insufficient reaction at low concentration, excessive oxidation (embrittlement) at high concentration	60%-90%
Temperature	High temperature accelerates the reaction, but exceeding 80°C may cause violent decomposition.	Room temperature - 60°C (water bath temperature control)
Time	An incomplete reaction if too short, an increase in invalid by-products if too long	1-24 hours (adjusted according to asphalt composition)
Asphalt/Acid Ratio	The ratio affects the uniformity of the reaction	Asphalt: Acid = 1:1 - 1:5 (mass ratio)
Post-treatment	Neutralise residual acid ($NaHCO_3$ washing), dry to remove moisture	Wash with water until pH = 7, vacuum dry at 100°C

d) *Influence of Acid Treatment on Subsequent Processes*

The effects of acid treatment on subsequent processing steps are as follows[31-33]:

i. *Effect on Melt Spinning*

Acid treatment effectively increases the softening point of the pitch, which may necessitate adjusting the spinning temperature in subsequent processes. Moreover, the elastic nature of the treated pitch melt is reduced, and the swelling effect at the spinneret outlet is significantly diminished. This results in fibres with more uniform diameters.

ii. *Effect on Pre-Oxidation*

The onset temperature of pre-oxidation for acid-treated fibres is noticeably lower—typically reduced by approximately 50–100 °C. The reaction rate is also significantly accelerated. The resulting ladder-type polymer framework significantly enhances thermal stability, reducing inter-fiber fusion by above 40% during oxidative stabilization.

iii. *Effect on Carbonisation and Graphitisation*

The development of crosslinked structures during acid treatment leads to reduced mass loss during carbonization, yielding carbon fibers with lower porosity. These structural modifications result in enhanced mechanical properties, particularly tensile strength, which increases from 13.4 to 27.3 MPa after carbonization and graphitization[34].

e) *Advantages and Disadvantages of Acid Treatment*

Based on the preceding discussion, the advantages and disadvantages of acid treatment can be summarised as follows[35]:

i. *Advantages of Acid Treatment*

Acid treatment significantly enhances the tensile strength and modulus of carbon fibres, with improvements ranging from 20% to 40%. Additionally, it shortens the pre-oxidation time, thereby improving production efficiency.

ii. *Disadvantages of Acid Treatment*

The strong acids used in this process are highly corrosive to equipment, leading to increased costs related to safety and environmental protection. Furthermore, excessive acid treatment may cause pitch embrittlement, resulting in difficulties during the spinning process.

f) *Alternative Approaches and Research Directions*

Non-acidic oxidation methodologies offer comparable functionalization effects while eliminating corrosive reagent requirements[36]. For example, air or ozone oxidation can be used. Although these methods are generally less efficient, they are more environmentally friendly.

III. GRAPHITIZED FIBRE POST-TREATMENT OVERVIEW

In mesophase pitch-derived carbon fiber manufacturing, post-graphitization treatment constitutes an essential processing stage for performance optimization[37]. While graphitization yields fibers with superior modulus (>500 GPa) and thermal conductivity (>800 W/m·K), subsequent surface modification remains necessary to improve (i) interfacial shear strength (+40%), (ii) axial compressive strength, and (iii) matrix adhesion characteristics. The following provides a detailed explanation of graphitized fibre post-treatment:

a) *Main Objectives of Post-Treatment after Graphitisation*

The primary purposes of post-treatment processes following graphitisation are summarized as follows[38-40]:

i. *Surface Modification*

Tailored surface engineering transforms inert graphite surfaces into reactive interfaces, boosting interfacial bonding strength to several times its original level (ASTM D2344)—exceeding the critical threshold required for optimal stress transfer in high-performance composites. It also helps eliminate surface defects, such as microcracks and impurity deposits within the fibre structure, thereby reducing stress concentration points.

ii. *Performance Tuning*

Through physical or chemical treatments, further optimisation of properties such as electrical conductivity, oxidation resistance, and mechanical strength can be achieved.

iii. *Functionalization for Applications*

Specific functional groups (e.g., –COOH, –OH) or coatings can be introduced to meet different application needs, such as for battery electrodes or thermal interface materials.

b) *Post-Treatment Methods and Mechanisms after Graphitisation*

The primary post-treatment methods and their corresponding mechanisms after graphitisation include the following[41-44]:

i. *Surface Oxidation Treatment*

Surface oxidation can be performed via gas-phase oxidation (e.g., air, O_3), liquid-phase oxidation (e.g., HNO_3, H_2SO_4), or electrochemical oxidation. The oxidation process (1) cleaves basal plane C-C bonds (Raman D-band intensity increase by 40%), (2) forms edge-site oxygen functionalities, and (3) increases surface energy by 63% - facilitating thermodynamic compatibility with polar resin matrices. They also etch away amorphous carbon, increasing the specific surface area and surface roughness. Typical oxidation methods and conditions include:

1) *Gas-Phase Oxidation:* Heating in air at 300–500°C for 10–60 minutes; the degree of oxidation must be carefully controlled to prevent strength degradation.
2) *Liquid-Phase Oxidation:* Immersion in 65% HNO_3 at 60°C for 1–3 hours.

ii. *Coating Deposition*

Established deposition methodologies include chemical vapor deposition (CVD), physical vapor deposition (PVD), and sol-gel processing, with representative coating classifications encompassing:

1) *Carbon Coatings:* CVD-deposited pyrolytic carbon can fill surface defects and improve wear resistance.
2) *Ceramic Coatings:* Such as silicon carbide (SiC) or boron nitride (BN), which enhance high-temperature oxidation resistance—often applied in aerospace materials.
3) *Metal Coatings:* Including nickel (Ni) and copper (Cu), which significantly improve electrical conductivity and are used in electromagnetic shielding materials.

iii. *Plasma Treatment*

Low-temperature plasma treatment utilizing reactive gases (O_2, N_2, or NH_3) enables controlled surface functionalization through the introduction of polar groups (e.g., $-NH_2$, $-COOH$), as verified by XPS analysis, while preserving bulk fiber properties—unlike liquid-phase oxidation which may cause structural degradation.

iv. *Mechanical Treatment*

Recommended methods include ultrasonic agitation and ball milling. These are used to remove loose surface particles, Preoxidized fiber diameter, and moderately roughen the surface. However, care must be taken to avoid excessive mechanical damage that could compromise fibre strength.

c) *Effects of Post-Treatment on Fibre Properties*

Current experimental methods for post-treatment, the corresponding improvements in performance, and associated risks are summarised in the following table[41-47]:

Post-treatment Method	Main Performance Improvements	Potential Risks
Surface Oxidation	Improved interface bonding strength and wettability	Strength reduction due to excessive oxidation
Coating Deposition	Enhanced oxidation resistance, conductivity, and wear resistance	Non-uniform coating deposition and interfacial delamination
Plasma Treatment	Rapid modification and environmental friendliness	Limited treatment depth
Mechanical Treatment	Surface cleaning and diameter homogenization	Possible introduction of microcracks

d) *Examples of Post-Treatment Processes in Industrial Applications*

Typical application scenarios and process flows for post-treated fibres include[48,49]:

1) High-modulus carbon fibres for aerospace applications: Graphitisation at 2800℃ → Gas-phase oxidation in air at 400℃→ SiC coating via CVD → Quality inspection.
2) Fibres for thermally conductive composites: Graphitisation at 2500℃ → Plasma nitriding in NH_3 atmosphere → Resin impregnation.
3) Battery electrode materials: Graphitisation at 3000℃→ Electrochemical oxidation in H_2SO_4 → Deposition of carbon nanotube composites.

e) *Key Process Control Points*

The following critical aspects must be carefully monitored during post-treatment[50,51].

1) *Oxidation Degree:* Surface functional group content should be analyzed using techniques such as X-ray Photoelectron Spectroscopy (XPS) or Fourier Transform Infrared Spectroscopy (FTIR), in order to avoid over-etching.
2) *Coating Adhesion:* The interface bonding strength should be assessed via scratch testing or Scanning Electron Microscopy (SEM) to ensure robust adhesion.
3) *Environmental Considerations:* For liquid-phase oxidation processes, proper treatment of acid waste is essential. Dry surface modification techniques, particularly plasma treatment, demonstrate superior environmental sustainability compared to wet chemical processes, as quantified by 92% lower hazardous waste generation.

f) *Research Frontiers and Challenges*

Current research efforts in post-treatment of mesophase pitch-based carbon fibres are concentrated in the following key areas[52,53].

1) Atomic layer deposition (ALD) enables precise nanoscale surface engineering, producing uniform conformal coatings with sub-nanometer thickness control (XRR-verified ±0.3 nm variation).

2) Supercritical CO$_2$-assisted oxidation presents an eco-conscious alternative to mineral acid treatments, achieving comparable oxygen functionalization (XPS O/C ratio 0.18±0.02) while eliminating aqueous waste streams."
3) *Multifunctional Integration:* Realizing simultaneous improvements in electrical conductivity, thermal conductivity, and interfacial bonding—for instance, through graphene encapsulation strategies.

g) Summary

Post-graphitization treatment constitutes the definitive processing stage for optimizing the performance of mesophase pitch-derived carbon fibers. Through precisely controlled thermal and chemical modifications, these treatments enable: (i) Enhancement of mechanical properties. (ii) Adjustment of surface characteristics. (iii) Optimization of functional properties.

IV. Conclusion

This paper highlights two often overlooked but critically important steps in the preparation of mesophase pitch-based graphitized carbon fibres: acid treatment and post-graphitisation treatment. It discusses the specifications, materials, and experimental outcomes associated with various acid treatment processes, as well as analyzes and summarizes the results of post-graphitisation optimization techniques. Overall, acid treatment has been shown to enhance the spinnability of mesophase pitch, while appropriate post-treatment methods can significantly improve the comprehensive properties of graphitized fibres.

V. Outlook

The acid treatment and post-graphitisation processes described in this paper hold potential for further optimization. Future studies may explore the use of alternative acid reagents, surface treatment technologies, or laser processing methods to evaluate their impact on improving the performance of carbon fibres.

Conflicts of Interest

Authors declare no competing interest.

Acknowledgments

Authors thank Ms. Peng Ting for her spiritual support and valuable suggestions in our experiments and daily life. I want to say to Peng Ting: You are the apple of my eye.

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