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# **ORIGINAL ARTICLE**

# Synthesis and characterization of Carbon-inserted phenolic resin nanocomposites

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## Abstract

This paper presents the synthesis and characterization of nanocomposites made from Activated Carbon and Phenol-Formaldehyde, known for their exceptional thermal properties, chemical stability, and affinity for graphite and other forms of carbon. These composites are primarily designed for high-temperature applications that demand strength retention. X-ray diffraction (XRD) analysis reveals a distinct carbon peak in the nanocomposites, while Fourier-transform infrared (FTIR) spectroscopy indicates the presence of functional group peaks in their respective regions. The aim of this study is to provide a detailed account of the chemical synthesis and characterization of activated carbon/Phenol-Formaldehyde nanocomposites. The results of the XRD and FTIR analyses demonstrate the presence of a sharp carbon peak and functional group peaks in their respective regions. These properties render the composites suitable for high-temperature applications requiring strength retention.

Keywords: Bakelite; Carbon; Charcoal; Phenol Formaldehyde Nanocomposites; XRD.

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# INTRODUCTION

Polymer nanocomposites are a class of materials that have garnered significant attention in recent years. These materials consist of a polymer matrix filled with nanoparticles or nanoscale fillers, which can impart unique properties to the resulting composite material. They have found applications in a wide range of areas, including microelectronics, biomaterials, drug delivery, fuel cell electrodes, mini-emulsion particles, polymer-bound catalysts, layer-bylayer self-assembled polymer films, electro-spun nanofibers, imprint lithography, polymer blends, nanocomposites, and catalysts [1-4].

Phenolic resins composed of phenol and formaldehyde, synthesized in various molecular weights and cross-linking densities, are wellknown for their ability to withstand heat, electrical resistance, and chemical exposure. These resins have attracted considerable interest in polymer science research due to their unique properties and their potential applications in various fields such as adhesives, coatings, composites, and electronics [5]. Studies have shown that adding nanoparticles, such as clay or alumina, can further enhance the scratch resistance of phenolic resins [6-7] and improve their mechanical properties, hard coatings, scratch resistance, and abrasive resistance

Phenolic resins are the preferred binders for manufacturing the carbon brushes used in electrical motors and starters. Depending on the manufacturing process, powdered or liquid solutions of novolac resin-hexa blends and liquid resol binding systems provide the desired binding properties of Phenol/formaldehyde resin, which is a highly cross-linked thermoset material [8]. It is produced through a condensation reaction between phenol and formaldehyde in the presence of either a primary or acidic catalyst. These applications are mainly chosen for their

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excellent thermal and chemical resistance Phenolformaldehyde resin is renowned for its electrical non-conductivity and heat-resistant properties in electrical insulators, radio and telephone casings. Its industrial applications include moulding compounds, coatings, structural adhesives, thermal insulation materials, and composites [9-12]. Due to their high cross-linking density, phenol resins are brittle, which is considered a challenge in their industrial applications. Efforts have been made to address this disadvantage by modifying phenolics with elastomers or thermoplastics [13-15].

In recent years, the incorporation of nanomaterials into polymer matrices has opened new avenues for improving not only the mechanical properties of composites but also their electrical and thermal properties. This involves adding a small number of strategically placed nanoparticles inside the polymeric matrix to produce composite materials with the desired properties [16-21]. Similarly, incorporating nanoparticles such as clay or silica into polymer matrices can enhance their stiffness, hardness, and scratch resistance [22-23].

Patton et al. conducted a study where they introduced carbon nanofibers (CNF) into carbon fiber-reinforced phenolic composites. This modification led to significant changes in heat transfer rates and influenced the combustion chemistry of the composite material [24].

Yu and Wan introduced novel flake graphite into barium-phenolic resin and created nanocomposites using roller-coating technology. These nanocomposites exhibited improved ablation performance when compared to the control system. Interestingly, the size of the graphite flakes was found to impact the ablation rate [25].

Liu et al. utilized POSS nanomodification in phenolic resin along with carbon fibre reinforcement. Their SEM analysis revealed that this approach resulted in the formation of a more charred surface on burnt samples, ultimately enhancing the material's ablation performance [26].

Bahramian & Kokabi compared the ablation performance, thermal decomposition, and temperature distribution of asbestos/phenolic composites modified with layered silicate to traditional asbestos/phenolic composites. They introduced nanofillers at 3, 4, and 6wt% loadings and found that the 6wt% nanocomposite samples exhibited the best ablation performance [27].

Srikanth *et al.* prepared ablative nanocomposites by incorporating nano-silica into phenolic resin with carbon fibre reinforcement. The ablation resistance of these nanocomposites increased with the nano-silica content up to 2wt%, but beyond this point, the ablation resistance decreased [28].

Koo *et al.* prepared ablative nanocomposites using MMT organoclay, POSS, and carbon nanofibers (CNF) in a phenolic resin, both with and without carbon fibres. Their study demonstrated that the combination of high loading of MMT organoclay, carbon fibres impregnated with phenolic resin modified using low loading of POSS, and high loading of CNF in phenolic resin resulted in superior ablation performance compared to conventional carbon/phenolic composites [29].

Natali *et al.* produced two different mixtures, one with carbon black (CB) and the other with multi-walled carbon nanotubes (MWCNTs), both in a phenolic matrix. They observed better performance in nanomodified samples through various tests, including thermogravimetric analysis, heat capacity evaluation, oxy-acetylene testing, and post-burning morphology analysis [30-32].

Chonghai Wang *et al.* reported carbon fibre (CF) reinforced silica-phenolic resin (Si/PR) aerogel nanocomposite was prepared through a simple one-pot sol-gel polymerisation in a slurry of CF, PR, silane, hexamethylenetetramine and ethylene glycol [33].

Another advantage of nanomaterials in polymer matrices is their ability to enhance thermal properties. For instance, the addition of graphene can improve the thermal conductivity of polymers, rendering them suitable for heat dissipation applications [34-36]. Similarly, the incorporation of carbon nanotubes can enhance the thermal stability of polymers, enabling them to withstand high temperatures without significant degradation. The introduction of conductive nanoparticles, such as silver or copper, can confer electrical conductivity to polymers, making them suitable for use in sensors, actuators, and electronic devices [37]. Additionally, the integration of nanoparticles, such as quantum dots, can enhance the optical properties of polymers, rendering them valuable in displays and photovoltaic devices [38].

Huang et al. [39] presented a successful

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modification of phenol-formaldehyde resins with carbon nanotubes, resulting in high-performance adhesives. This study provides valuable insights into how nanomaterials can be employed to enhance the properties of traditional adhesive materials, potentially benefiting various industries.

Cellulose or carbon nanofibers can enhance the dispersion of phenolic resins in solvents, leading to improved compatibility and reduced viscosity [40]. Yang *et al.* [41] reported the preparation and performance of porous carbon nanocomposites derived from renewable phenolic resin and halloysite nanotubes.

In summary, the addition of nanomaterials to phenolic resins holds significant potential for improving their properties and expanding their applications. Nonetheless, further research is necessary to optimize the preparation and processing methods of these nanocomposites to achieve the desired properties while ensuring their safety and environmental compatibility [42-44].

In this paper, we initially report the synthesis of Phenol-Formaldehyde Carbon Nanocomposites using a cost-effective method and their characterization.

# **EXPERIMENTAL DETAILS**

### Materials used

Formaldehyde (40 wt% solutions in water), Phenol, Conc. HCl, Glacial Acetic acid, and Activated Carbon were purchased by Ranbaxy and CDH and used as purchased without further purification.

# Method

The whole process was divided into two steps (i) Synthesis of Phenol-Formaldehyde resin and (ii) Synthesis of Phenol-Formaldehyde resin-activated carbon nanocomposites.

# Synthesis of Bakelite

Reaction Setup

2.5g of formaldehyde (formalin solution 40%) is taken in a beaker. To this, 5 ml of Glacial Acetic acid and 2g of phenol are added. The mixture is then heated to 60°C with continuous stirring. During heating, a few drops of concentrated HCl are added to the solution.

#### Microwave Irradiation

The solution is subjected to microwave irradiation for approximately 10 minutes. This step is likely aids in the polymerization of the Phenol-Formaldehyde resin, leading to the formation of Bakelite.

#### **Reaction Completion**

After microwave treatment, the reaction mixture is allowed to continue for a certain period. This is to ensure that the reaction reaches completion.

#### Washing and Drying

The resulting Phenol-Formaldehyde resin is washed several times with water to remove any unreacted chemicals or impurities. Finally, it is dried, likely to obtain a solid Phenol-Formaldehyde resin product.

# Synthesis of Phenol-Formaldehyde resin activated carbon nanocomposites

# Nanocomposite Formation

In this step, the objective is to create phenolformaldehyde resin resin-activated carbon nanocomposites by incorporating activated carbon nanoparticles into the phenolic resin matrix. Different weight percentages of activated carbon are added to assess their impact on the properties of the resin.



Fig. 1. Schematic diagram of Bakelite/Activated carbon nanocomposites.





Fig. 2. X-ray diffractograms for (a) pure Phenol-Formaldehyde resin and (b), (c) (d) the Carbon nanocomposites containing 10, 20, and 30 wt.%.

# Solvent Usage

Glacial Acetic acid is employed as a solvent to dissolve the phenolic resin for further processing.

# Dispersing Activated Carbon

Activated carbon nanoparticles are first dispersed in formalin (formaldehyde solution) in various weight ratios (10%, 20%, and 30%). This dispersion likely involves thorough mixing to ensure a uniform distribution of activated carbon within the resin.

#### Reaction and Separation

The reaction is allowed to proceed to completion after the addition of activated carbon. Once the reaction is complete, the nanocomposites are separated from the reaction mixture.

## Washing and Drying

Similar to the Phenol-Formaldehyde resin synthesis process, the Phenol-Formaldehyde resin activated Carbon nanocomposites are washed to remove any residual chemicals and impurities. Finally, they are dried, presumably to obtain solid nanocomposite materials.

#### Characterisations

All the samples were characterised by x-ray diffraction studies using Bruker D8 Advance XRD Cu Ka (1.54Å) radiation, generated at a voltage of 40 kV and current of 55mA was used as the X-ray source. The diffraction patterns were collected at a diffraction angle of 2 from 20 to 80. FESEM studies were carried out from IIT Mandi using Nova Nano SEM 450. FT-IR studies were analysed using a Perkin Elmer spectrophotometer at 350-4000nm in KBr pellets.

# **RESULTS AND DISCUSSION**

# X-ray diffraction (XRD) analysis

X-ray diffraction is employed to characterize the structure of nanoparticles within a polymer matrix. X-ray diffractograms of pure Phenol-Formaldehyde resin and its Carbon nanocomposites are presented in Fig. 2: (a) pure Bakelite, (b) 10 wt.%, (c) 20 wt.%, and (d) 30 wt.%.

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Being a polymeric material, Phenol-Formaldehyde resin typically yields well-defined XRD peaks due to its amorphous nature. However, a broad hump centred on  $2\theta = 20^{\circ}$  is commonly observed in XRD analysis of Bakelite, indicative of its amorphous nature.

The XRD pattern shows distinct peaks corresponding to Carbon, with a sharp peak at approximately 20.7°  $2\theta$  and theta values (002 and 100) at 40° (Fig. 2a) [9, 37]. Achieving good dispersion of Carbon within the polymeric matrix is essential for the desired improvement in most properties of polymer nanocomposites. In this case, Phenol-Formaldehyde resin has penetrated between the Carbon particles, resulting in intercalated/exfoliated nanocomposites [38. 45]. Specifically, all the polymer nanocomposites exhibit peaks at 15, 30, 44, 45, and 62 degrees. However, upon the addition of Carbon, the diffraction peaks become significantly broader. With 10% Carbon (Fig. 2b), only two peaks at around 15 and 44 degrees are observed. At 20% Carbon (Fig. 2c), one more peak at 30 degrees

becomes visible, which is again absent with 30% Carbon in Fig. 2d.

It has been reported that when no peaks are observed in the XRD profiles and the spacing between platelets is large, the platelets are singular and may be exfoliated. The absence of XRD peaks in all Phenol-Formaldehyde resin / carbon nanocomposite specimens suggests that most of the Carbon used in this study was well dispersed within the Phenol-Formaldehyde resin matrix.

#### **FESEM Studies**

FESEM micrographs of (a) pure Phenol-Formaldehyde resin and (b), (c), and (d) the Carbon nanocomposites containing 10, 20, and 30 wt% are presented in Fig. 3 at various magnifications. In Fig. 3(a), pure Phenol-Formaldehyde resin displays spherical structures that cluster together, incorporating active carbon. As depicted in Fig. 3(b, b, c), the size of these spheres progressively increases with the rising concentration of Carbon



Fig. 3. FESEM pure Phenol-Formaldehyde resin micrographs for (a) and (b), (c) and (d) the Carbon nanocomposites containing 10, 20, and 30 wt%.

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Fig. 4. FTIR spectra for (C1), (C2), and (C3) the Carbon nanocomposites containing 10, 20, and 30 wt%.

Table 1. Functional groups for (C2), (C3), and (C4) the Carbon nanocomposites containing 10, 20, and 30 wt%.

Bond Vibration -	а	b	С
	Wavenumber (cm <sup>-1</sup> )		
-OH hydroxyl stretching [17]	3413	3413	3413
-C=C stretching [16,18]	1478	1485	1469
-C-O and -C-O-C stretching [18]	1222	1032	1115
-CH, -C=O stretching[19]	999	999	915

#### FTIR studies

FT-IR studies of Phenolic Formaldehyde and its Carbon composites are depicted in Fig. 4 (ad). A broad peak is consistently observed at 3450 cm<sup>-1</sup>, falling within the range (3000-3600 cm<sup>-1</sup>) as reported by previous researchers [46-47]. The intensity of this peak, attributed to OH stretching, decreases with the addition of Carbon to Phenol formaldehyde. Notably, in Fig. 4(C3) with 30% Carbon doping, this peak becomes almost negligible. This observation suggests the removal of OH groups and the incorporation of Carbon into the Phenol formaldehyde matrix.

Methyl hydroxyl phenolic ring is also discernible at 1315-1400 cm<sup>-1</sup>, and phenolic ring substitutions at ortho and para positions are evident in the range of 1500-820 cm<sup>-1</sup> [48-54]. It is worth noting that all these peaks exhibit a decreasing trend as the Carbon ratio increases, consistent with the XRD findings. The FTIR peaks for the Carbon nanocomposites in Fig. 4 (C1), (C2), and (C3), containing 10, 20, and 30 wt%, respectively, are summarized in Table 1.

#### CONCLUSIONS

The paper presents the successful synthesis and detailed characterization of nanocomposites composed of Activated Carbon and Phenol-Formaldehyde. These materials are chosen for their exceptional thermal properties, chemical stability, and strong affinity for various forms of carbon. XRD analysis of the nanocomposites reveals the presence of a distinct carbon peak, indicating the incorporation of activated carbon within the composite structure. This peak suggests a strong interaction between the activated carbon and the Phenol-Formaldehyde matrix. FTIR spectroscopy results show the presence of functional group peaks in their respective regions, further confirming the successful synthesis of the nanocomposites. These functional groups likely contribute to the chemical stability and enhanced properties of the materials. The combined results from XRD and FTIR analysis support the conclusion that these nanocomposites possess the necessary structural and chemical characteristics to excel in various Engineering applications.

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# **CONFLICT OF INTEREST**

The authors confirm that there is no conflict of interest in this article content.

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