Journal Homepage: -www.journalijar.com

INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR)

ISSN (O): 2320-5407
ISSN (P): 3107-4928

Article DOI: 10.21474/IJAR01/23418
DOI URL: http://dx.doi.org/10.21474/IJAR01/23418

RESEARCH ARTICLE

INFLUENCE OF COPPER OXIDE NANOPARTICLES ON UV AGING RESISTANCE AND THERMAL DEGRADATION OF SULFONATED POLY (1, 4-PHENYLENE ETHER ETHER SULFONE) MEMBRANE

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Manuscript Info

Manuscript History
Received: 4 March 2026
Final Accepted: 8 April 2026
Published: May 2026

Key words:-
PEES, SPEES, copper oxide, thermal stability, optical studies

Abstract

The purpose of this study is to synthesize multifunctional membranes based on poly(1,4-phenylene ether ether sulfone) (PEES). PEES has good thermal, optical, electrical, and antimicrobial properties. Copper oxide nanoparticles reinforced in a PEES matrix. These reinforced membranes have a unique structure and properties that influence the thermal stability and UV protection compared with neat PEES. The PEES chain structure and composition of the copper oxide were shown to be associated with the degradation behavior. Since PEES is a hydrophobic polymer, it does not have good interaction with copper oxide nanoparticles. The resultant polymer chain was functionalized with H_2SO_4 to improve the hydrophilic nature and better dispersion of copper oxide nanoparticles in PEES. The concentration of copper oxide nanoparticles was varied in the sulfonated PEES matrix. The weight loss upon degradation was positively correlated with the concentration of copper oxide nanoparticles. The concentration of nanoparticle ratio varied, which dramatically changed the UV shielding properties of PEES. Copper oxide has potential electron correlation effects, high-temperature superconductivity, spin dynamics, and antimicrobial properties. Therefore, the sulfonated poly (1,4-phenylene ether ether sulfone) (SPEES) copper oxide membrane has improved thermal and optical properties.

Introduction:-

The simplicity, energy efficiency, and high separation operation of membrane technology have made it a critical platform in dealing with the most critical issues in water purification, biomedical engineering, and energy conversion systems. The classical membranes are mostly tailored to size exclusion or diffusion-based selective separation, but suffer from these problems: fouling, weak selectivity, and not being robust enough to work in severe conditions. In an effort to overcome these shortcomings, the idea of multifunctional membranes has been of considerable interest in the past few years. Multifunctional membranes are novel materials that have been designed to incorporate multiple functionalities in a single membrane system, allowing them to go beyond traditional

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separation processes. Such membranes can be engineered to possess more than one property, including antimicrobial response, catalyzed degradation, increased hydrophilicity, mechanical stability, thermal stability, and stimuli-responsiveness. Such a wide range of functionalities is not only capable of enhancing the effectiveness of the membrane but also increasing the duration of operation by reducing fouling and degradation. Multifunctional membranes have been used in water treatment systems with combined separation and degradation properties, so it is possible to remove organic contaminants, heavy metals, and pathogenic microorganisms. These membranes are also under investigation as wound dressing materials, drug delivery systems, and implant coating in the biomedical field because of their biocompatibility and antimicrobial properties. Moreover, multifunctional membranes have importance in energy applications, including proton exchange membranes in fuel cells, to enhance ion transport without damaging the structure in high-temperature and oxidative condition.

A polymer-based high-performance membrane with excellent optical, thermal, chemical resistance, electrical, and antimicrobial properties. These multifunctional properties have made an excellent place in material science. The synthesis of polymer nanocomposites is relevant due to the global rise in lightweight, durable, and high-efficiency materials in sectors such as electronics, biomedical engineering, energy storage, and environmental protection from temperature, ultraviolet rays, microbial infections, nosocomial (hospital-acquired) bacteremia, and the growing issue of antibiotic resistance. A primary focus has been on polymernanocomposites that has not only excellent physical properties such as hydrophilicity and thermal stability but also UV-resistant membranes. Biomaterial demand is increasing day by day in human life due to various factors such as accidents and lifestyle. Poly(1,4-phenylene ether ether sulfone) (PEES) is a thermoplastic polymer that has madeits place among high-performance polymers. It has excellent thermal stability, excellent film-forming properties [1], high proton conductivity, mechanical strength[2], antimicrobial properties,and resistance to chemicals [3]. These attributes make poly(1,4-ether ether sulfone) (PEES) an excellent choice in various fields, especially in filtration [3], and electronics [4]. High-performance coatings, artificial hearts, pacemakers, blood tubes, and various devices are used in medical science for those joints, bone plates, antibacterial coatings, and medical devices [5-6].

Poly(1,4-ether ether sulfone) (PEES)is a material that has an aromatic backbone; two ether and sulfone linkages provide moderate polarity with limited inherent hydrophilicity. The hydrophilic behavior of PEES can be modified through chemical modification by various acidssuch as sulfur trioxide, acetyl sulfate, and sulfuric acid, which promote improved surface wettability and water affinity. Such enhancement in hydrophilicity is animproved applicability of PEES in membrane and biomedical systems where effective interaction with aqueous environments is required [7-9]. Recently, copper oxide nanoparticles have shown a wide range of applications, such as catalytic systems, sensing devices, gas-sensing technologies, and pollution degradation, due to their resistance to photocorrosion, high oxygen-carrying capacity, and electrical and optical andsolar energy transformation. Further studies on it as an antibacterial, antiviral, anti-inflammatory, and antifungal agent for application for both biomedical and environmental uses [10-11].

The properties of copper oxide nanoparticles primarily depend on the size of the nanopowder, their morphology, the specific surface area of the prepared materials, and the synthesis methods. When these nanoparticles are used as filler in a polymer matrix, they enhance polymer matrix properties. The thermal and optical properties, including their ability to emit, absorb, and scatter light, depend upon copper oxide nanoparticle size and dispersion into a polymer matrix. Copper oxide and SPEES may combine the qualities to create a new material with unique physical and chemical properties [12]. In this study, we focus on the ethanol-based solution casting method. Functional and morphological studies were done by using FI-IR and SEM, and thermal degradation of the polymer nanocomposite was investigated by the help of TGA and DSC. We found that SPEES-copper oxide nanomaterial has better thermal degradation, UV resistance, and glass transition temperature (Tg) than neat SPEES.

Materials and Methods:-

Pure poly (1,4-phenylene ether ether sulfone) was obtained from Sigma-Aldrich. Copper oxide nanoparticles synthesized by Vedayukt India private limited, Jamshedpur, India. Synthetic grade dichloromethane (Purity>99.9%) and sulfuric acid (H_2SO_4) (Purity>99.9%) were purchased from Merck, India.

Sulfonation of pure poly (1,4-phenylene ether ether sulfone):-

Dichloromethane was used as a dissolving agent for poly (1,4-phenylene ether ether sulfone) pellets. PEES-dichloromethane mixture was stirred again washed with methanol to get PEES in a powdered form and dried at 60 ^{o}C during 12 h. H_2SO_4used as sulfonation agent and stirred with conc. H_2SO_4 during 5h and precipitated in cold deionized water. Sulfonated PEES was washed several times in cold deionized water and dried in 60 ^{o}C at 12 hrs [5].

Nanocomposites film formation:-

Sulfonated poly (1,4 phenylene ether ether sulfone)(SPEES) and copper oxide nanoparticles were combined indiffer ent amounts like 2%, 4%, and 6% by weight in table 1. The ethanol solution casting method was used to help the nanoparticles better dispersation. Copper oxide nanoparticles were mixed with ethanol and this mixture was sonicate d for 30 to 50 minutes to spread the nanoparticles evenly in the SPEES matrix. This sonicated mixture was then added to the SPEES ethanol solution.

The mixture was stirred continuously for 5 hours. Just before pouring itina petri dish,the solution was sonicated agai n for 3 minutes to stop the nanoparticles from clumping together and to keep them evenly spread in the SPEES matrix. The resulting membrane was then dried at 70 degrees Celsius.

Table 1. Loading concentration of copper oxide nanoparticles in SPEES matrix

Sample Code	Copper Oxide Loading (%)
SPEES	00
SPEES/CuO-01	2
SPEES/CuO-02	4
SPEES/CuO-03	6

Results:-

FT-IR spectra in the 400 to 4000 cm^{-1} range were used to characterize the functional groups that were present in thesulfonated poly (1,4-phenylene ether ether sulfone)(SPEES) and SPEES/copper oxide membranes. Figure 1 shows the -SO_3H group of H_2SO_4 and O-H stretching was observed at 3363 for neat SPEES that is shift 3383 and 3382 for the nanocomposite membrane. A sharp peak of C–C aromatic bond stretching was observed at 1583 cm^{-1}in the case of SPEES copper nanocomposite.1634 Sharpe peak for neat SPEES, but its intensity reduces in polymer nanocomposite.A peak at 553 cm^{-1} confirmed the presence of Cu-O. A peak at 1042 shows the confirmed presence of S=O stretching for SPEES that shifted to 1103 for nanocomposite membrane. The long polymeric chain was bending in the polymer, as evidenced by a peak of 869 cm^{-1}.

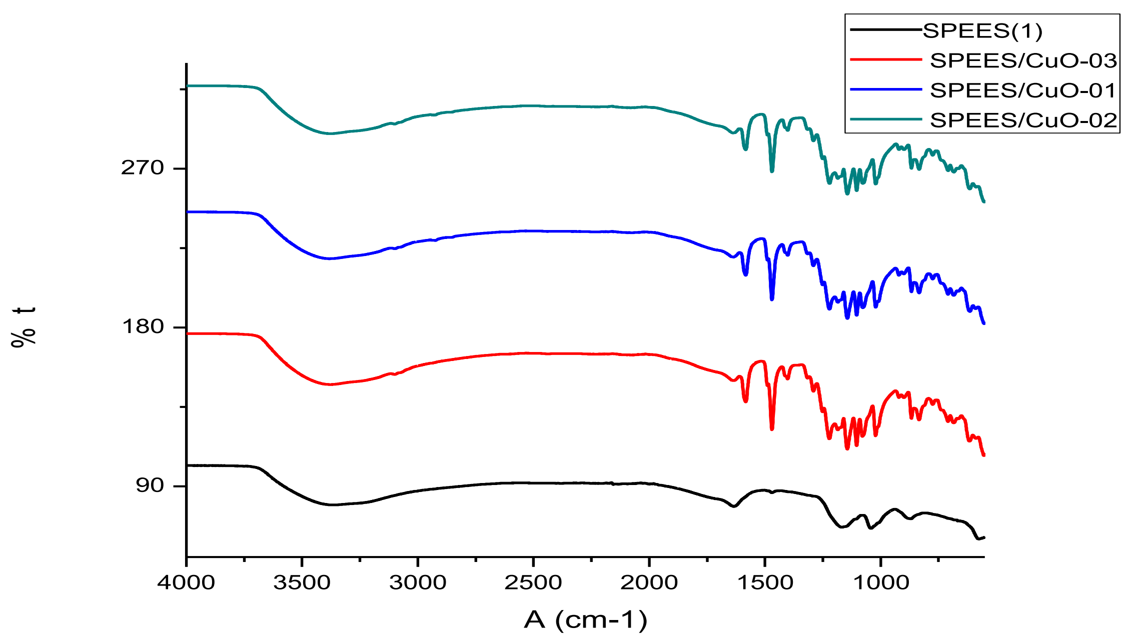

Figure1 FT-IR spectra of sulfonatedPEES and SPEES/copper oxide membranes

The surface morphology of the SPEES/copper oxide nanocomposite was studied by SEM. It explains the shape, size, dispersion, and compatibility of nanoparticles in a polymer matrix. Good dispersion of a filler in a polymer matrix significantly improves composite performance by improving thermal properties, optical properties, and antibacterial properties. Figure 2 shows that copper oxide nanoparticles have a pellet form and are well dispersed into the sulfonated poly(1,4-phenylene ether ether sulfone) (SPEES) matrix. It shows good interaction between SPEES and copper oxide nanoparticles.

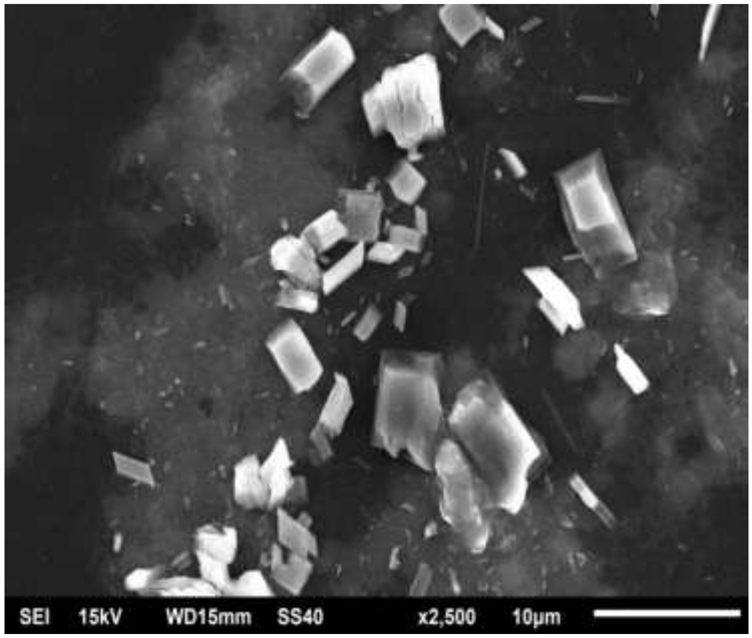

Figure 2.SEM image of SPEES/copper oxide membrane

Thermal analysis is a technique that provides information about how polymer material decomposes with heat. Figure 3 shows sulfonated poly (1,4-phenylene ether ether sulfone)(SPEES) and SPEES/copper oxide membranes start losing weight due to heat occurring at the temperature of 150-300°C. This weight loss occurs due to the removal of absorbed water molecules or solvent. The second stage, located at 300–450°C, resulted from the sulfonic acid groups in SPEES and SPEES-copper oxide in the ion exchange polymer membranes. The third weight loss started at 450°C onwards due to the degradation of polymerbackbones.

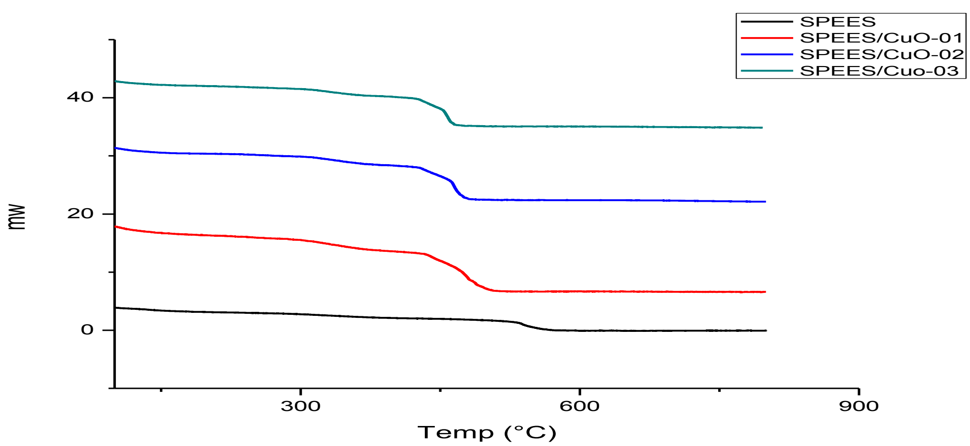

Figure 3. Comparative TGA graph of sulfonatedPEES and SPEES/copper oxide membranes

DSC analysis gives details about the glass transition temperature (Tg) and melting point of polymer. Tg determined that transition from a hard, brittle, glassy state to a soft, rubbery state. Figure 4 shows that copper oxide nanoparticles were well dispersed in SPEES matrix, and the resultant glass transition temperature (Tg) was increased with copper oxide nanoparticle increase in neat SPEES. 6% copper oxide loading has more Tg than 2%, 4%, and neat SPEES.

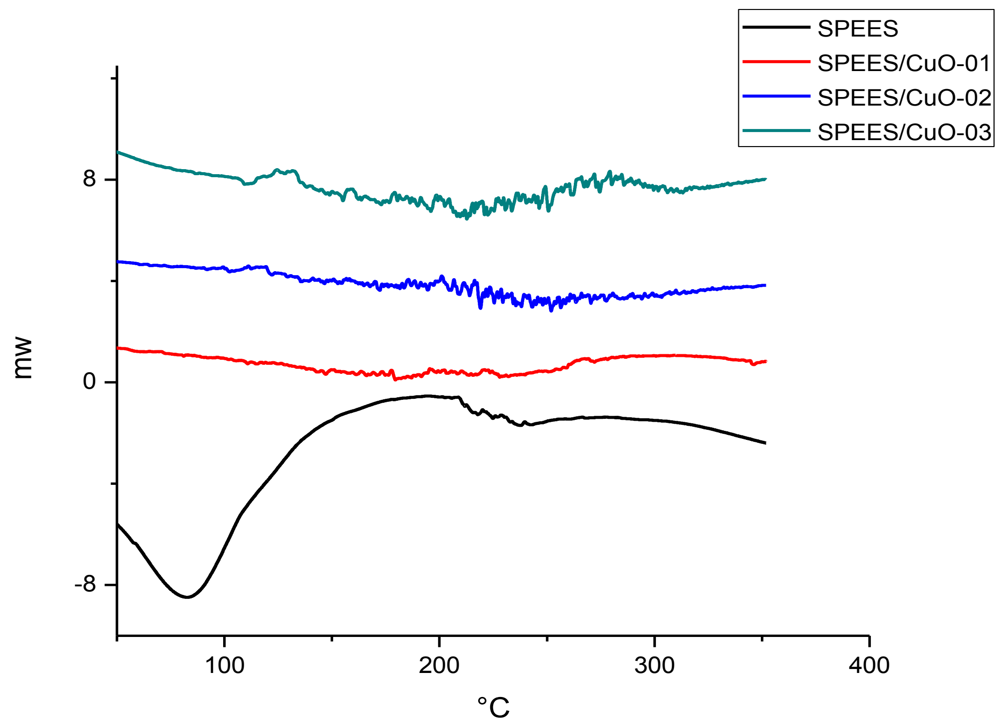

Figure 4. Comparative graph of glass transition temperature (Tg) of sulfonated PEES and SPEES/copper oxide membrane

UV-absorption lies between 250 to 300 nm. Figure 5 shows the absorption of SPEES and SPEES/copper oxide membranes. A slight change was observed in the UV absorption of copper oxide. Absorption of SPEES/copper oxide membranes showed a higher absorption peak intensity increase than that of neat sulfonated poly (1,4-phenylene ether ether sulfone)(SPEES). 2% and 4%loading did not have much effect on the absorption peak, but 6% copper oxide shifted the absorption peak.

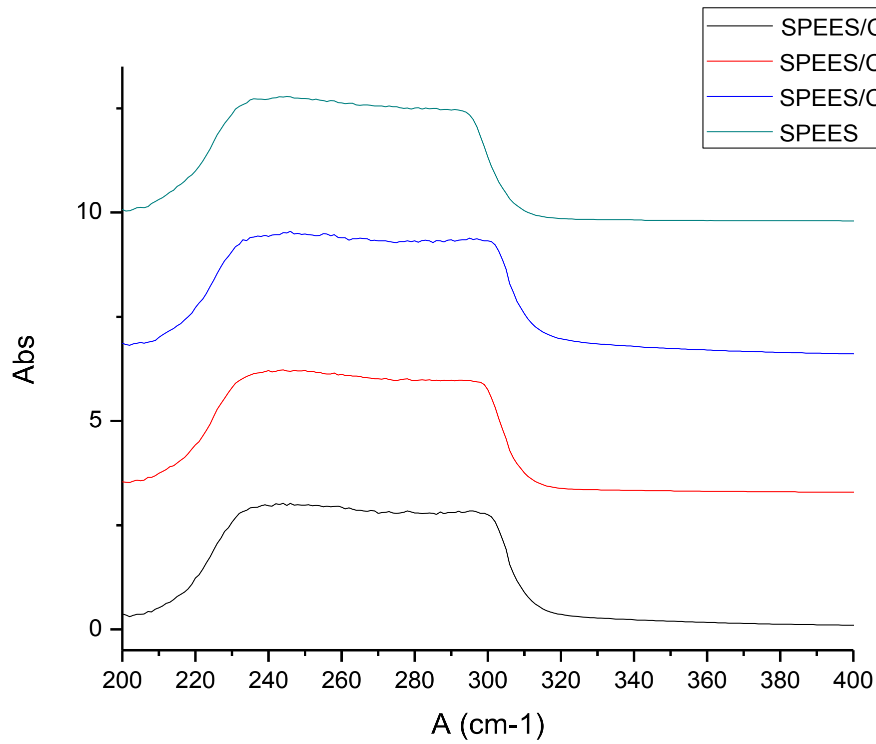

Figure 5. Comparative graph of UV Spectra of sulfonatedPEES and SPEES/copper oxide membranes

Discussion:-

The characteristic C–O C stretching vibration in the neat SPEES backbone, initially observed at 1167 cm^{-1}, shifted to 1144 cm^{-1} and 1185 cm^{-1} upon the incorporation of copper oxide (CuO) nanoparticles. These shifts confirm a significant chemical interaction between the CuO fillers and the SPEES matrix. Thermal stability enhanced progressively with CuO loading; specifically, the degradation onset temperature shifted toward higher values compared to the neat SPEES (Table 2). This improvement is attributed to the high degree of nanoparticle dispersion and robust interfacial adhesion, which effectively restricts polymer chain mobility. Consequently, the thermal resistance of the composite is significantly bolstered. Notably, the residual char yield decreased as CuO loading increased, suggesting that these nanocomposites are better suited for high-temperature applications.

Table 2 Thermal degradation of sulfonatedPEES and SPEES/copper oxide membranes

Sample Code	Onset-point	Mid-point	End-Point	Final Weight loss percentage
SPEES	342	485	554	100
SPEES/CuO-01	359	420	488	99.8
SPEES/CuO-02	429	438	471	100
SPEES/CuO-03	447	449	472	99.5

Furthermore, the addition of CuO nanoparticles led to an increase in the glass transition temperature (Table 3),further confirming the reduction in chain segmental mobility. Optical analysis (Figure 5) revealed an absorption peak at 244 nmfor neat SPEES, which underwent a bathochromic shift to 282 nm in the nanocomposites. This red shift serves as evidence of strong interfacial interaction and uniform filler dispersion, ultimately enhancing the optical properties of the SPEES-CuO system.

Table 3 Glass transition temperature (Tg) of sulfonatedPEES and SPEES/copper oxide membranes

Sample Code	Glass transition temperature (Tg)
SPEES	82.7
SPEES/CuO-01	91
SPEES/CuO-02	100
SPEES/CuO-03	105

Conclusion:-

This study demonstrates that the incorporation of copper oxide (CuO) nanoparticles significantly enhances the thermal stability of sulfonated poly(ether ether sulfone) (SPEES). Specifically, the 6% loading exhibited superior thermal properties compared to both neat SPEES and the 2% and 4% loadings. This improvement is attributed to the excellent dispersion and strong interfacial interaction of the CuO nanoparticles within the SPEES matrix due to the improvement in the hydrophilic nature of PEES. A 6% copper oxide nanoparticle concentration has a better glass transition temperature than 2% and 4% concentrations. Furthermore, the 6% loading enhanced UV absorption relative to the pristine polymer.

CRediT authorship contribution statement:-

Anjali Chhonkar: Writing –original draft, Methodology, Investigation, Visualization Data curation, and Conceptualization. Harikant: Supervision, and administration. Gautam Jaiswar: Investigation, and Writing –review & editing.

Acknowledgment:-

The authors wish to thank Jiwaji University, Gwalior, and SAIF, Aligarh Muslim University, analysis on a paid basis.

Funding detail:-

We do not have any financial support for this research.

Declaration of interest statement:-

There are no relevant financial or non-financial competing interests to report.

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