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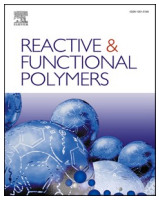

Functionalized polymer & metal oxide nanocomposite material for efficiency antibacterial and photocatalytic applications

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ABSTRACT

Functionalized polymer&metal oxide nanocomposite materials are distinguished by their multifunctional properties. In this work, for the synthesis of a functional polymer-metal oxide nanocomposite material, polyvinyl chloride (PVC) was first modified with polyethyleneimine (PEI) under heterogeneous conditions. After amination, the Cu (II) ions were adsorbed onto anion-exchange (PPE-4) material. The resulting polymer/metal complex was then thermally treated to synthesize a functional nanocomposite CuO&PPE-4 material containing copper oxide nanoparticles on the polymer surface. Synthesized functional CuO&PPE-4 nanocomposite material was characterized using UV–Vis, PL, FTIR, Raman, XRD, SEM-EDX, and BET surface analysis to identification its structure, morphology, and physico-chemical properties. The antibacterial activity was tested against *Escherichia coli (E.coli)* and *Pseudomonas aeruginosa (P. aeruginosa)* for Gram-negative as well as *Staphylococcus aureus* (*S. aureus*) for Gram-positive bacteriums. Revealing a 26 ± 0.5 mm inhibition zone for *E. coli,* 35 ± 0.5 mm for *P. aeruginosa* and 21 ± 0.5 mm *S. aureus* which significantly exceeded that of Cu^{2+}&PPE-4 due to enhanced reactive oxygen species (ROS) generation and improved charge separation. Moreover, the photocatalytic degradation of the functional CuO&PPE-4 material of tetracycline (TC) was evaluated under sunlight degradation. UV–Vis spectroscopy confirmed a progressive decline in TC absorbance at 276 nm and 358 nm, indicating effective photocatalysis. The process followed pseudo-first-order kinetics, with rate constants of 1.01685 min^{-1} (5 mg/L TC), 0.90951 min^{-1} (10 mg/L), and 0.48637 min^{-1} (20 mg/L). Possible reaction pathways for the photocatalytic degradation of TC are presented based on HPLC MS analysis. The functionalized CuO&PPE-4 nanocomposite material synthesized at 150 °C exhibited a low band gap of 1.53 eV than other materials, which ensured effective photocatalytic and antibacterial activities. Furthermore, practical results show that the functionalized CuO&PPE-4 nanocomposite material removes TC very effectively from pharmaceutical industry wastewater.

1. Introduction

In the last decade, interest in multifunctional polymer materials has been growing at an upward trend. Multifunctional polymer materials have a wide range of applications due to their unique physicochemical properties due to the presence of chemically active functional groups and metal oxide nanoparticles in the macromolecules [1]. Materials with such unique properties are currently widely used in various

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industries. Such as catalisys [2], wastewater treatment [3], optoelectronics [4], pharmaceutics [5], biomaterials [6], medicine [7], building materials [8], coatings [9], and many more applications rely on multifunctional polymers. It is observed that the introduction of metal oxide nanoparticles into a polymer material containing an active functional group not only stabilizes the nanocrystals, but also enables the functional group and nanocrystals to exhibit new functions. In particular, nanocomposite materials obtained by incorporating copper oxide nanoparticles into functional polymers are distinguished by their unique functionality. Multifunctional nanocomposite materials containing copper oxide nanoparticles are widely used in environmental protection, medicine, pharmaceuticals, food packaging, surface coatings, and other fields due to their antibacterial, catalytic, photocatalytic, and biocompatible properties [10,11]. Although physical, chemical and biological methods have been used to remove heavy metal ions and pollutants from wastewater, these approaches are often ineffective in chemical and biological treatment processes.

Polycomplexes of chitosan [12], polymethacrylate [13], polyaniline [14], polyvinyl chloride [15] and other polymers have been used as sorbents for water treatment. However, their filtrates have limited susceptibility to biological treatment, so a combination of two or more materials must be used to overcome these obstacles [16]. Therefore, the development of polymeric composite materials with high adsorption properties and different functional groups is crucial. Metal oxide nanoparticles are significant reactivity, huge surface area, pore size, particles shape, heat resistant and chemically stable as well as have high antibacterial and photocatalytic activity. Nevertheless, agglomeration during their synthesis can reduce their unique properties [17]. Polymer stabilizers with adsorption properties are used to prevent this phenomenon. In the synthesis of polymer-metal oxide nanoparticles, a number of functional synthetic and natural polymers are used as a polymer matrix that stabilizes metal oxide nanocrystals.

Functional materials containing copper oxide (CuO NPs) nanoparticles are widely used in environmental protection due to their unique physicochemical properties. For example, they are widely used in the food industry and medicine to remove harmful microorganisms from wastewater [18,19], and in the agricultural [20], pharmaceutical [21], and textile industries [22] to remove toxic organic compounds from wastewater. Functional nanocomposite materials containing CuO NPs are synthesized using modern nanotechnology methods. In this study, the authors prepared CuO NPs on chitosan surface by biosynthesis method. The biosynthesized functional nanocomposite material exhibited effective antibacterial and cytotoxic properties against breast cancer [23]. The authors synthesized a nanocomposite material by incorporating CuO NPs onto the surface of a functional polymer obtained by graft polymerization of N-ethylpiperazine in polyvinyl chloride using green synthesis [24]. The synthesized nanocomposite material demonstrated high antibacterial properties against *E. coli* and *S. aureus* bacteria. Also, in this study [25], polymer nanocomposites with copper oxide by effective antibacterial properties against *E. coli* and *S. aureus* bacteria were synthesized. In line with the above studies, in this study, an antibacterial material against *E. coli*, *S. aureus* and Salmonella bacteria was synthesized by hydrothermally forming CuO NPs on cellulose surfaces [26]. In this study, the authors reported that the synthesized functional material, CuO NPs on chitosan surface, using a green synthesis method from environmentally friendly *Ficus carica* leaf extract, exhibited effective antibacterial properties against several bacteria [27].

The chemically active functional groups in the polymer matrix of multifunctional nanocomposite materials containing copper oxide nanoparticles (CuO NPs) contribute to the sorption properties of the material and the stabilization of CuO nanocrystals, as well as to the antibacterial, cytotoxic, biocompatible, catalytic, and photocatalytic properties of the nanocrystals. The authors [28] synthesized a functional nanocomposite containing CuO NPs by modifying polydopamine with CuO, for which they applied CuO oxide nanoparticles to polydopamine using a hydrothermal method. It was observed that the synthesized nanocomposite material exhibited higher antibacterial properties compared to other polymer composites containing CuO nanoparticles. This is explained by the enhanced antibacterial properties of CuO NPs as a result of the action of amine and hydroxyl groups in the functional polymer and the defect-free formation of nanocrystals. Also, in this study [29], they developed an effective antimicrobial agent against bacterial cells by forming copper oxide and silver nanoparticles on the surface of Gum Arabic. CuO NPs were synthesized by the authors using a green synthesis method on the surface of silk fibroin [30]. The synthesized functional material efficiently (91.95 %) photocatalytically decomposed methylene blue textile dye under UV light. This study [31] found that the nanocomposite material obtained by incorporating CuO NPs into the antibiotic had higher antibacterial properties than the antibiotic.

Currently, wastewater from the pharmaceutical industry is increasingly contaminated with various harmful antibiotics. Due to their high toxicity, potential mutagenicity, and carcinogenicity, they have a serious negative impact on human, animal, and environmental health, and therefore the removal of harmful antibiotics from pharmaceutical wastewater is an important research issue [32,33]. The adsorption method is considered ineffective for cleaning pharmaceutical wastewater from harmful antibiotics, since the problem of eliminating the adsorbed antibiotic after desorption is not completely solved. Therefore, photocatalytic degradation of antibiotics to a harmless state using functional photocatalysts containing metal oxide nanoparticles is an effective method. Among antibiotics, tetracycline (TC) occupies a large proportion of the drugs use and is frequently employed for disease treatment and growth promotion in animal husbandry [34,35]. Photocatalytic degradation involves the use of semiconductor materials, such as metal oxides (NiO, ZnO, TiO_2, or CuO), that, under visble irradiation, generate electron-hole (e/h) pairs. In this case, the lifetime of photogenerated photoelectrons from the metal oxide is short. To extend the lifetime of photogenerated photoelectrons, semiconductor metal oxide nanoparticles are formed on the surface of various functional polymer matrices. In this case, the photogenerated electrons in conduction band of semiconductors can immigrate to polymer matrics that significantly prevent from e/h recombination [36–39]. These pairs produce reactive oxygen species (ROS), such as hydroxyl radicals (•OH) and superoxide radicals ($\bullet O_2^-$), which oxidize and degrade complex organic molecules like tetracycline into simpler, non-toxic compounds. The efficiency of this process depends on factors like the photocatalyst's bandgap, surface area, and stability, as well as environmental conditions such as pH, light intensity, and pollutant concentration [40–42]. With such properties, based on functional materials containing metal oxide nanocrystals, TC degradation to harmless substances has been carried out, including the $NiFe_2O_4/CeO_2$/GO functional material synthesized by the authors, which photocatalytically degraded TC in aqueous media with an efficiency of 95 % [43]. The authors [44] also synthesized a functional black titanium oxide-zinc oxide nanocomposite material via the sol-gel method and used it to remove TC from wastewater. The results of the study showed that the black titanium oxide-zinc oxide material photocatalytically decomposed TC with 63 % efficiency under sunlight. Baoum and Amin [45], synthesized graphene oxide (CuO/GO) supported on CuO NPs by sol-gel synthesis. This synthesized photocatalyst was used to photocatalytically decompose TC in the presence of visible light to purify wastewater. However, there are no studies conducted so far on the influence of chemically active groups in the functional polymer matrix on the properties of semiconducting CuO NPs and their participation in charge transport during the decomposition of organic pollutants.

Nowadays, wastewater from pharmaceutical industries is purified from harmful antibiotics and microorganisms using sorbents through the sorption process. Though the sorption purification method turned out to be ineffective, and on the other hand, the problem of eliminating harmful antibiotics and bacteria released during the regeneration of the sorbent after sorption remains unresolved. The main novelty and

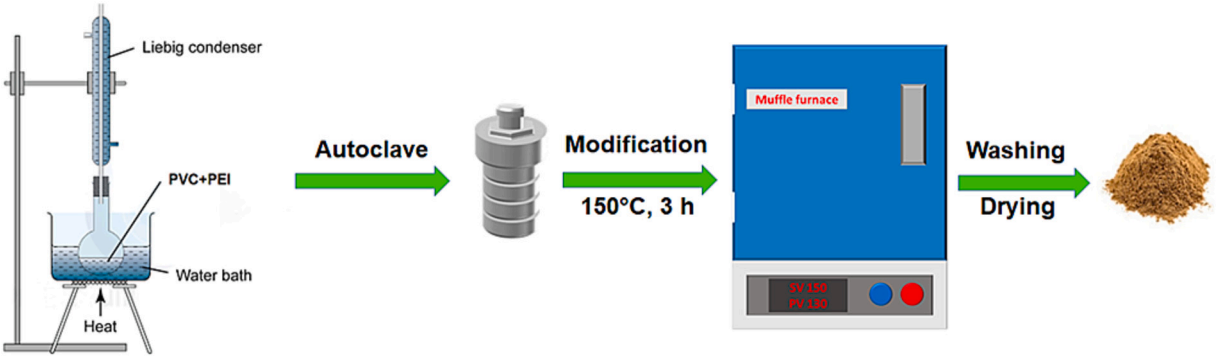

Fig. 1. Schematic representation of the sinthesys aminated polyvinylchloride.

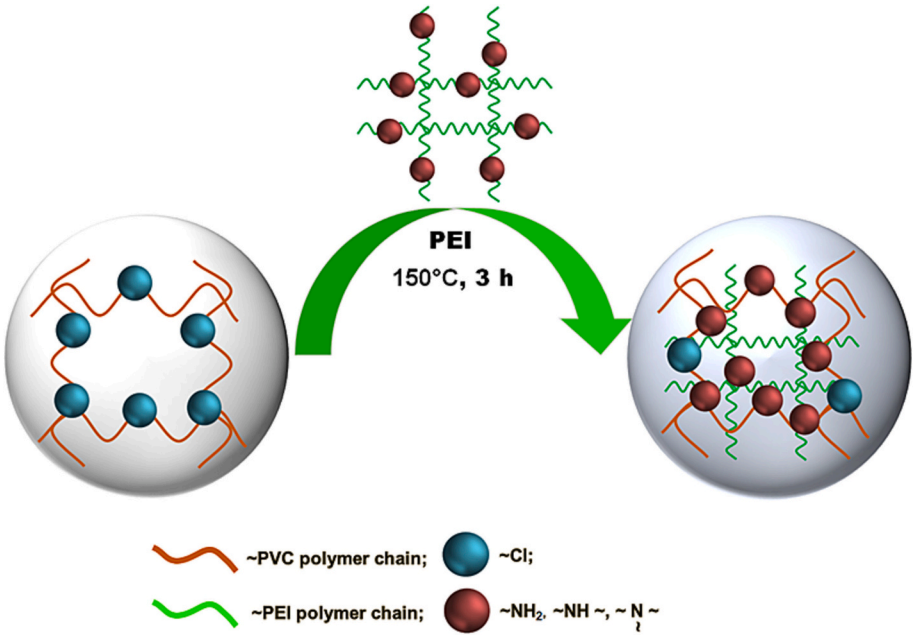

Fig. 2. Reaction scheme of modification of the polyvinylchloride with polyethylenimine.

importance of this study was the antibacterial and photocatalytic properties of a multifunctional (CuO&PPE-4) nanocomposite material obtained by forming copper oxide (CuO) nanoparticles on the surface of aminated polyvinyl chloride (PPE-4) were investigated. The structural and morphological characteristics of the synthesized multifunctional CuO&PPE-4 nanocomposite materials were identified using various techniques. The narrower bandgap energies of the synthesized functional CuO&PPE-4, Cu^{2+}&PPE-4, and PPE-4 materials are 1.53 eV, 1.74 eV, and 2.28 eV, respectively. It was found that the surface area of the functional CuO&PPE-4 (85.3 m^2/g) nanocomposite material was twice as large as that of the PPE-4 (42.6 m^2/g) material. The optical activity and surface area of the functional CuO&PPE-4 material, compared to other synthesized functional materials, influenced the manifestation of superior antibacterial and photocatalytic properties. The antibacterial properties are tested against *Pseudomonas aeruginosa* (*P. aeruginosa*) *Escherichia coli* (*E. coli*) for Gram-negative and *Staphylococcus aureus (S. aureus)* for Gram-positive bacteriums. The antibacterial properties of the synthesized multifunctional CuO&PPE-4 nanocomposite material are 1.5 times better than those of the Cu^{2+}&PPE-4 material. The synthesized functional CuO&PPE-4 material completely degraded the antibiotic TC in a solution with a concentration of 200 ppm in 240 min. In addition, with the increase in the concentration of TC antibiotic in the solution to 50 ppm, 100 ppm, 200 ppm, the photocatalytic decomposition rate constant decreased to 1.016 min^{-1}, 0.909 min^{-1}, 0486 min^{-1}. Fursemore, the CuO&PPE-4 nanocomposite material exhibited high antibacterial and photocatalytic properties when the formation temperature was 150 °C and the concentration of Cu^{2+} ions in the solution was 0.1 mol/L.

2. Materials and methods

2.1. Materials

All reagents used in this study were of analytical purity. The anion exchanger PPE-4 was synthesized in-house by modifying powdered polyvinyl chloride (PVC, ≥99 % purity, particle size <100 μm, density 1.38 g/mL, JSC "NAVOIAZOT", Navoi, Uzbekistan) with branched polyethyleneimine (PEI, ≥99 % purity, molecular weight 10,000 Da, viscosity 200–500 mPa·s at 20 °C, Sigma-Aldrich). Sodium hydroxide (NaOH, ≥98 %, pellets) and hydrochloric acid (HCl, 36 %, ACS reagent grade) were employed to activate the PPE-4 anion exchanger, sourced from Sigma-Aldrich. Copper(II) chloride dihydrate ($CuCl_2{\cdot}2H_2O$, ≥99 %, trace metals <0.01 %), ammonium hydroxide (NH_4OH, 25–30 % NH_3 basis), and isopropyl alcohol (≥99 %, HPLC grade) were also obtained from Sigma-Aldrich for the synthesis of CuO&PPE-4 nanocomposites. Deionized water (DI, resistivity ≥18 MΩ·cm, at pH = 7, 25 °C) was

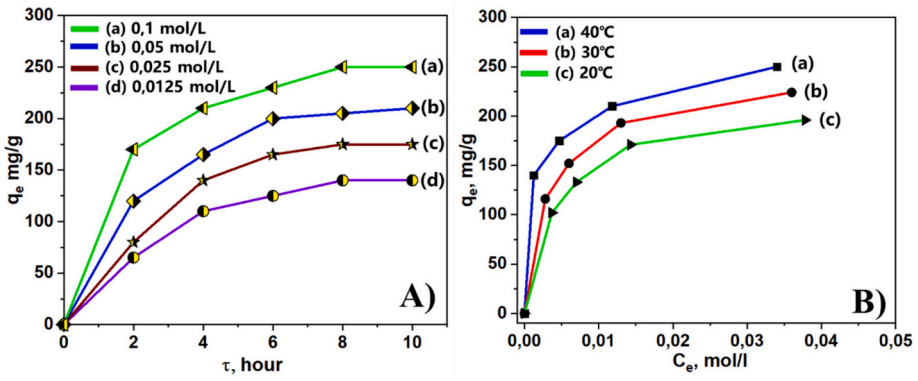

Fig. 3. Sorption kinetics (A) and isotherms (B) of Cu^{2+} ions on the anion exchanger PPE-4.

produced using a Heal Force water purification system (Shanghai, China) and used throughout the experimental procedures.

Bacterial strains *Escherichia coli (E. coli)* (MTCC 443, gram-negative), *Pseudomonas aeruginosa (P. aeruginosa)* (MTCC 1688, gram-negative) and *Staphylococcus aureus (S. aureus)* (MTCC96 Gram-positive) were acquired from the Microbial Type Culture Collection and Gene Bank (MTCC, Chandigarh, India) for antibacterial testing. Miller-Hinton agar (dehydrated, TM Media, New Delhi, India) was used as the growth medium. Tetracycline hydrochloride (TC, ≥95 %, USP grade, Sigma-Aldrich) served as the target compound for photocatalytic degradation studies. All materials were stored under conditions recommended by the suppliers (e.g., TC at 4 °C in darkness, chemicals at room temperature) to maintain stability and purity during the experiments.

2.2. Synthesis of PPE-4

The PPE-4 anion exchanger was synthesized through a two-step chemical modification process involving the reaction of polyvinyl chloride (PVC) with polyethyleneimine (PEI) to introduce amine functionalities, adapting established protocols [46–50]. Industrial-grade PVC powder (3.0 g) was combined with 10 mL of 70 % (*w/v*) aqueous PEI (≥99 %) in a 100 mL round-bottom flask equipped with a magnetic stir bar. The mixture was stirred at 300 rpm and heated in a water bath at 80 °C for 2 h, initiating partial substitution of PVC's chlorine atoms with PEI's amine groups, with full modification achieved during subsequent hydrothermal treatment. The reaction mixture was transferred to a 50 mL Teflon-lined stainless steel autoclave to achieve complete modification and enhance the crosslinking density. The autoclave was sealed and placed in a muffle furnace, where it was heated to 150 °C for 3 h under autogenous pressure (approximately 4–5 bar, estimated from water vapour pressure at this temperature). Optimal synthesis conditions were determined as 150 °C for 3 h based on prior optimisation studies, balancing reaction completion with the thermal stability of the polymer components. After the hydrothermal treatment, the autoclave was cooled to room temperature over 2 h in air. The resulting PPE-4 solid was recovered by vacuum filtration through a filter paper using a Buchner funnel and a vacuum pump. The solid was washed with 200 mL of deionized water in four 50 mL aliquots to remove unreacted PEI and residual salts, followed by drying in a vacuum oven at 90 °C for 24 h under a reduced pressure of 10 mbar.

The static ion exchange capacity of the synthesized PPE-4 was determined titrimetrically by equilibrating 0.5 g of the dried material with 50 mL of 0.1 mol/L HCl for 24 h, followed by back-titration with 0.1 mol/L NaOH using phenolphthalein as an indicator. The measured capacity was 5.5 mg-eq/g, confirming the successful incorporation of amine groups into the PVC matrix. The synthesis process is schematically represented in Fig. 1, and the reaction scheme illustrating the substitution of chlorine with PEI's amine groups is depicted in Fig. 2.

2.3. Adsorption studies

Adsorption studies were conducted to evaluate the Cu^{2+} ion uptake capacity of the PPE-4 anion exchanger under controlled conditions, determining adsorption kinetics and equilibrium isotherms to optimize subsequent CuO&PPE-4 synthesis. Copper (II) chloride dihydrate ($CuCl_2{\cdot}2H_2O$) solutions (0.0125, 0.025, 0.05, and 0.1 mol/L) were prepared from a 1 mol/L stock solution by dissolving 17.045 g of $CuCl_2{\cdot}2H_2O$ in 100 mL deionized water and diluting in 25 mL volumetric flasks. The pH was adjusted to 6.0 (±0.05) using 0.1 mol/L NaOH or HCl, monitored with a pH meter, to ensure Cu^{2+} solubility. PPE-4 (0.3 g) was weighed and added to 100 mL of each Cu^{2+} solution in 250 mL conical flasks. The mixtures were stirred at 100 rpm in a temperature-controlled incubator (IKA KS 3000i control, Germany) at 20 °C, 30 °C, or 40 °C for 2, 4, 6, 8, or 10 h, using separate flasks for each time point and condition. After each interval, suspensions were filtered through 0.45 μm nylon syringe filters and residual Cu^{2+} concentrations were measured at 800 nm via UV–Vis spectroscopy (Shimadzu UV–1900i, Japan). Experiments were performed in triplicate, reporting mean values.

Adsorption capacity at time t (q_t, mg/g) was calculated using Eq. (1):

$$q_t = \frac{(C_0 - C_t)}{m} \times V \quad (1)$$

where C_0 is the initial Cu^{2+} concentration (mg/L), C_t is the concentration at time t (mg/L), V is the solution volume (0.1 L), and m is the PPE-4 mass (0.3 g). Equilibrium capacity (q_e, mg/g) was determined after 10 h using Eq. (2):

$$q_e = \frac{(C_0 - C_e)}{m} \times V \quad (2)$$

where C_e is the equilibrium concentration (mg/L). Maximum sorption capacity reached 248 mg/g at 30 °C and 0.1 mol/L, defining optimal CuO&PPE-4 synthesis conditions (30 °C, 0.1 mol/L, 10 h) [51]. Kinetic and isotherm data are presented in Fig. 3A and B, respectively.

The sorption kinetics of copper ions by the obtained anion-exchange material based on PVC/PEI at different concentrations and durations of sorption (See Fig. 3(A)). In the Fig. 3(A) shows that with increasing duration of the process, an increase in specific sorption occurs. In the first two hours, sorption is directly proportional to the duration of the process. Thereafter, the rate of the process slows down, and equilibrium is achieved after approximately 10 h. In the, Fig. 3(B) shows the sorption isotherms of Cu^{2+} ions on the PPE-4 anion exchangers at different temperatures. An increase in the concentration of Cu^{2+} ions and an increase in the temperature of the initial solution leads to increased sorption. Based on these results, the best conditions for synthesizing the functional CuO&PPE-4 nanocomposite were determined to be a temperature of 30 °C, a copper ion concentration of 0.1 mol/L, and a sorption duration of 10 h. The anion exchanger material synthesis

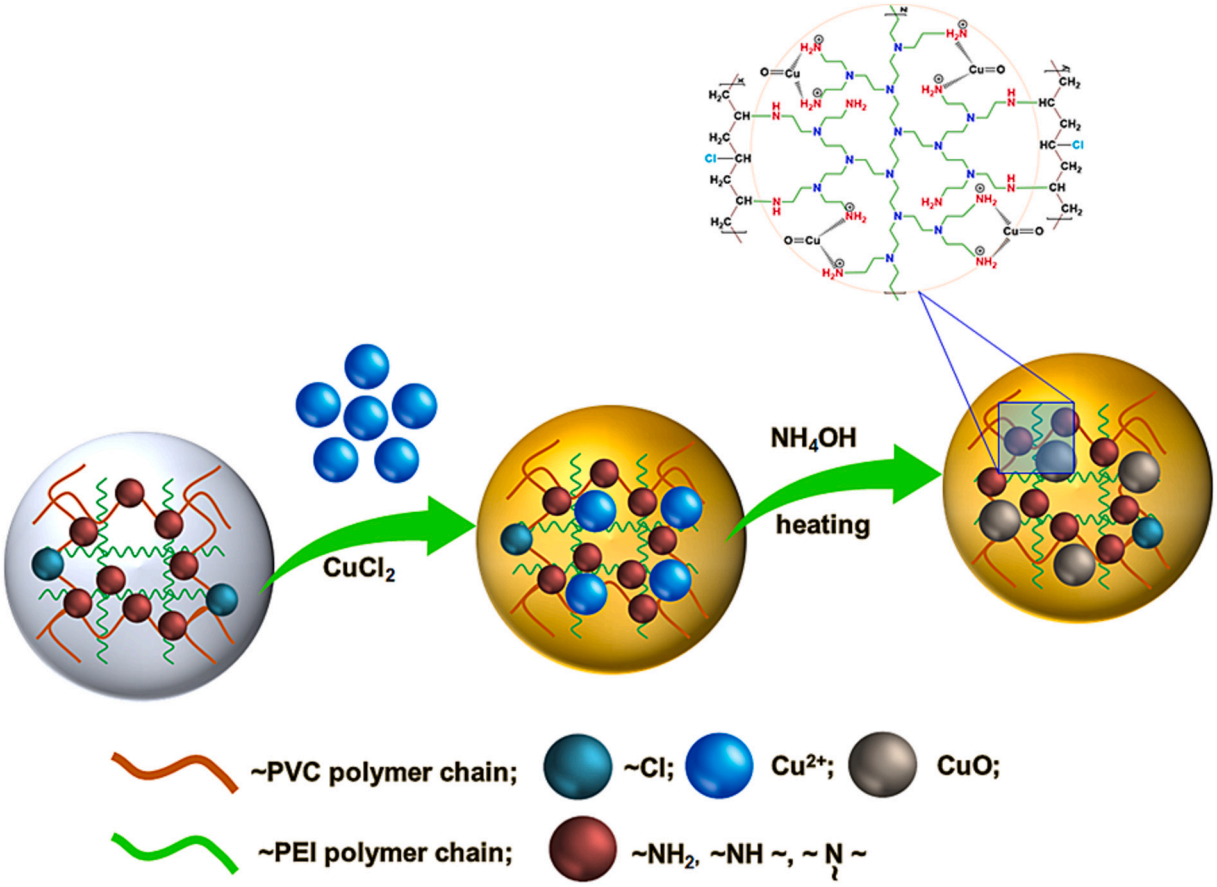

Fig. 4. Schematic representation of the formation of functional CuO&PPE-4 material.

scheme is described below: the anion exchange material obtained based on PVC/PEI contains ~chloro and ~ amino functional groups. During the sorption of copper ions onto the anion exchange material, the copper ions bind to the ~chloro and ~ amino functional groups through coordination bonds. The anion exchange material PPE-4, synthesized from PVC/PEI, exhibited a maximum copper ion sorption capacity of 248 mg/g through the formation of these coordination bond (See Fig. 4).

In order to further investigate the sorption process of Cu (II) ions on sorption material PPE-4, the sorption isotherms of PPE-4 were measured to investigate the sorption capacity of the sorption material at different initial concentrations of Cu (II) ions containing solution. As a result of studying the sorption of Cu (II) ions to PPE-4 sorption material at different temperatures (20, 30 and 40 ^{0}C) (See Fig. 3(B)), the sorption of Cu (II) ions increased with the increase in the temperature of the sorption medium. At a temperature of 40 ^{0}C, the absorption value reached a maximum. This can be explained by the increase in the activity of ions in the solution with the increase in temperature and high internal diffusion. Therefore, the optimal temperature for the sorption process was taken as 40 ^{0}C.

The Langmuir and Freundlich models were used to study the sorption patterns of Cu (II) ions onto the PPE-4 sorption material. The Langmuir and Ferundlich constants were calculated using the data in Figs. 3-A and B above, and these data are presented in Table 1 above. According to the isotherm constants, the value of the Freundlich isotherm constant ***n*** is greater than 1, which indicates the high efficiency of the absorption of Cu (II) ions to the sorption material. The proximity of the Langmuir isothermal correlation coefficient to 1 indicates that the sorption of Cu (II) ions into the sorption material proceeds by Langmuir isotherm laws [52,53]. In the Langmuir isotherm model, the value of R_L indicated the shape of the isotherm: unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < RL < 1$) and irreversible ($R_L = 0$). The values of RL were found to be 0.986 in this study anion exchanger PPE-2 resin, indicating favorable sorption of Cu (II) by anionic resins. From the results of the sorption isotherm, we can conclude that Cu (II) ions are maximally adsorbed at a temperature of 40 ^{0}C, 10 h and a concentration of Cu (II) ions of 0.1 mol/L. Also, during the sorption process, Cu (II) ions form a complex with the amino groups of the PPE-4 sorbent through a coordination bond.

2.4. Preparation of functional CuO&PPE-4 nanocomposite

The functional CuO&PPE-4 nanocomposite was synthesized by integrating CuO nanoparticles into the PPE-4 anion exchanger through a sequential coprecipitation and hydrothermal treatment process. PPE-4 (3.0 g) was dispersed in 50 mL of a 0.1 mol/L copper (II) chloride dihydrate solution, prepared by dissolving 17.045 g of $CuCl_2{\cdot}2H_2O$ in deionized water. The mixture was stirred at 100 rpm and maintained at 40 °C (±2 °C) for 10 h in a 100 mL flask to facilitate Cu^{2+} ion exchange with the amine groups of PPE-4, forming an intermediate Cu^{2+}&PPE-4 complex. Subsequently, a 1 mol/L ammonium hydroxide solution was added dropwise (1 mL/min) at 60 °C until the pH reached 8–9, as measured by a pH meter, inducing a colour change from pale blue to dark green, signifying $Cu(OH)_2$ precipitation within the polymer matrix.

The reaction mixture was then divided into three equal portions and transferred to 50 mL Teflon-lined autoclaves. Hydrothermal treatment was conducted under three conditions to optimize CuO nanoparticle formation: 110 °C for 6 h, 130 °C for 8 h, or 150 °C for 10 h, with temperatures controlled within ±5 °C. After cooling naturally to room temperature for over 2 h, each product was filtered through a 0.45 μm nylon membrane filter, washed sequentially with 100 mL DI water and 50 mL isopropyl alcohol to remove unreacted reagents, and dried in a vacuum oven at 90 °C under 10 mbar pressure for 24 h. The resulting functional CuO&PPE-4 nanocomposite exhibited well-dispersed CuO