Gamma Irradiation-Induced Preparation of Polyacrylonitrile Acrylamide Nano-Silica for Removal of Some Hazardous Metals

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Research Article

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Posted Date: August 19th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-809665/v1>

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Version of Record: A version of this preprint was published at Journal of Inorganic and Organometallic Polymers and Materials on November 17th, 2021. See the published version at <https://doi.org/10.1007/s10904-021-02156-1>.

Gamma irradiation-induced preparation of polyacrylonitrile acrylamide nano-silica for removal of some hazardous metals

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Abstract

Gamma-irradiation initiated polymerization was utilized to prepare polyacrylonitrile acrylamide nano-silica {P(AN-AM)-NS}. Various analytical tools like XRD, FT-IR, SEM, TEM and DTA & TGA were used to estimate the morphology, functional groups, and structure of {P(AN-AM)-NS} nanocomposite. The ability of {P(AN-AM)-NS} nanocomposite to remove Cs(I), Pb(II), Cd(II), Sr(II), and Cu(II) ions from the multi-component system was evaluated by batch techniques considering the influence of (shaking time, pH, reaction temperatures), and capacity. At the optimum pH, distribution coefficients have selectivity order; $Pb^{2+} > Cs^{+} > Cu^{2+} > Cd^{2+} > Sr^{2+}$. The kinetic data obey pseudo-second-order models. The capacity was reduced by increasing the heating temperatures of solid powder. The thermodynamic parameters showed an endothermic and spontaneous. The investigation proved that {P(AN-AM)-NS} nanocomposite is a suitable organic-inorganic sorbent for the sorption of the studied ions from liquid solutions and could be considered as potential material for purification of effluent polluted with these ions.

Keywords: Gamma-irradiation. Polymerization. Nano-silica. Distribution Coefficients. Kinetic. Thermodynamic.

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1 Introduction

Resin exchangers for environmental pollution remediation were widely used by different investigators [1–3]. Many organic & inorganic sorbents were reported with wide-ranging utility. Organic sorbents have impoverished thermal and radiation stability and are therefore unstable in strong radiation fields at high temperatures [4]. Inorganic exchangers are very stable towards high radiation and temperatures but are inadequate to treat huge volumes of radwaste and costly [5]. Thus, to solve restrictions related to both organic & inorganic sorbents, efforts were made for the combination of composite sorbents with enhanced properties [6]. Composite sorbents were produced by mixing polymers as supporting materials with inorganic species as an exchanger. The material is mechanically, thermally, and chemically more stable [7,8]. The polymer in composite material improves the mechanical, optical characteristics and provides more exchangeable spots [9]. The composite sorbents had a strong interest due to their variation of usages in unlike fields such as water purification, chemical separation, electrochemical sensor, solar cell, optical properties, and catalyst [9,10]. Many pollutants disrupt or change the chemical structure of water in the world and affect the aquatic environment [11]. The current methods for the sorption of hazardous metals from liquid waste solutions include chemical precipitation, ion exchange, membrane, adsorption, and biosorption [11]. Recently it is one of the biggest challenges to remove contaminants from wastewater, as their importance increases with growing industrial activities [2]. Studies on % uptake of Pb(II), Cs(I), Cu(II), Sr(II), & Cd(II) ions from liquid media have been focused largely on adsorption and ion exchange methods [12–14]. The potential toxicity and adverse health effects are shown by toxic metals such as Cd, Cu, Pb, Cs, and Sr ions.

In current work, polyacrylonitrile acrylamide nano-silica {P(AN-AM)-NS} was fabricated using the gamma radiation technique at 50 kGy. This material was characterized by utilizing various analytical tools. The main objective of the current work is to prepare nanocomposite and explore the efficiency of this nanocomposite towards the sorption of Cs(I), Pb(II), Cu(II), Sr(II), & Cd(II) ions from liquid waste.

2 Experimental

2.1 Chemicals and reagents

The main chemicals used to prepare {P(AN-AM)-NS} were Na₂SiO₃ (Loba Chemie, India), acrylonitrile (AN), acrylamide (AM), N, N methylene bisacrylamide, and dimethyl ketone (α -Chemika, India). Chemical such as CsCl, CuCl₂.2H₂O, SrCl₂, PbCl₂, CdCl₂, AgNO₃, HNO₃, and HCl (Merck, Germany)

and KOH (El-Nasr, Egypt). Chemicals utilized in this article have an analytical assessment. For all tests, preparing (sample & solutions) double-distilled water (DDW) was employed. Measurements of the initial pH of studied metal ion solutions were completed utilizing a lab pH-meter, (pH 601A, (USA).

2.2 Gamma cell

A Co⁶⁰ γ -cell of class MC-20 (Russia), was applied as a radiation foundation for the polymerization process at the Cyclotron, Egypt. The solution was irradiated in a plastic bottle. The irradiation dosage rate was ~473.35 Gy/h.

2.3 Preparation

2.3.1 Fabrication of nano-silica (NS)

As reported [15], NS was fabricated by addition of concentrated HCl (dorpwith) to Na₂SiO₃ solution with constant stirring at (25±1 °C) until pH approximately (8–9), gel of silica was obtained, and aged for one day, then decantation was carried out to separate the precipitate from the filtrate, accompanying washing with warm DDW sometimes until free from Cl⁻ was tested using AgNO₃ (dilute solution). After the drying at 80 °C, the NS powder collected, white NS were ground for 300 s in fast mills.

2.3.2 Preparation of {P(AN-AM)-NS}

At 25±1 °C and constant stirring, add drop by drop of 10 % (AN, AM & NS) solutions and 0.1 % N, N-methylene-bisacrylamide was developed for the production of {P(AN-AM)-NS} with volumetric ratios (AN: AM: NS) equal unity. The solution was agitated for 120 min to overcome complete homogeneity then subject to γ -irradiation within 50 kGy of about 4.38 days. After the complete radiation process, gel yield was cut into minor wreckages, soaking in dimethyl ketone for the elimination of unreacted components, rinsed with DDW, dried at 60 °C. The solid material was transformed into the hydrogen form by treating it for 24 h with 0.1 M HNO₃. The gel product was purified numerous times with DDW to confiscate the excess HNO₃ and dried at 60 °C.

2.4 Instruments

The studied material was analyzed with an IR spectrophotometer (Alpha II Bruker, Germany). XRD measurement is conducted (Shimadzu, Japan). The prepared nanocomposite was analyzed for TGA&DTA in N₂ atmosphere by a (Shimadzu DTG-60 H). Direct observation via SEM examined the

micrographs of the sample produced (model JSM-5400, JEOL Instrument, Japan). TEM was done using (JEM2100, Jeol. s.b, Japan).

2.5 Stability to different solvents

The stability of {P(AN-AM)-NS} to chemical attack was conducted by shaking 100 mg of nanocomposite and 100 mL of DDW, HNO₃, HCl, and KOH in a range of concentrations [1-6 M]. The solution with irregular shaking for about 4 days. The precipitate establish was detected gravimetrically [14].

2.6 Adsorption investigations

2.6.1 Influence of shaking time

An equilibrium was done by shaking 0.1 g of solid in H⁺ form with 10 mL of the multi-component system (Pb(II), Cs(I), Cu(II), Sr(II), & Cd(II) ions) in a shaker thermostat (Kottermann, Germany) at 25±1 °C with batch factor (V/m = 100 mL/g). After each time break, a shaker is fixed and the filtrate is removed at once from the solid. Hence, AAS was utilized to detect the tested ions concentration. The % removal can be known by the next equation [16];

$$\% \text{Removal} = \left(\frac{C_i - C_f}{C_i} \right) 100 \quad (1)$$

In which: C_i (initial concentrations) & C_f (final concentrations) of metal ions in solution.

2.6.2 Ion-exchange behavior

To examine the sorption of the multi-component system (Cs(I), Pb(II), Cu(II), Sr(II), & Cd(II) ions) using batch techniques, a sequence of experiments was applied at 100 mg/L for investigated cations at changed pH and 25±1°C. Both distribution coefficient (K_d) and separation factor (α_b^a) as a function of pH were calculated with the following equations [12]:

$$K_d \text{ (mL / g)} = \left(\frac{C_i - C_f}{C_f} \right) \frac{V}{m} \quad (2)$$

$$\alpha_b^a = \frac{K_d(b)}{K_d(a)} \quad (3)$$

where V and m are the solution volume (L), and {P(AN-AM)-NS} weight (g), a & b are two competing types in a system.

The kinetic analysis for the investigated cations onto the {P(AN-AM)-NS} in H⁺ form was taken place by applying the solid with investigated cations (100 mg/L) with a V/m ratio of 0.1 L.g⁻¹ at the wanted pH in a shaker thermostat at 25±1°C.

The influence of different reaction temperatures (25-65 °C) on the amount of sorbed ions (q). In each experiment, 10 mL of investigated metal ions at the preferred pH, and 0.1 g of solid in H⁺ form was vigorously shaken for 4 h using a thermostatic water bath shaker. The solution's pH has been adjusted with NaOH and/or HCl for all investigations.

2.6.3 Capacity measurements and thermal stability

Batch equilibration repeated for investigated cations (0.2 g/L) with the solid sorbent in V/m = 100 mL/g was applied for the capacity estimation at different heating temperatures (50-400°C) in a muffle kiln for 4 h. The multi-component systems (Cs(I), Pb(II), Cu(II), Sr(II), & Cd(II) ions) were agitated in a shaker thermostat at pH=4.3 for 4 h. The capacity was calculated from equation (4) [17,18].

$$\text{Capacity} = \sum \text{Uptake} \times C_i \times \frac{V}{m} \quad \text{mg / g} \quad (4)$$

3 Results & Discussion

The object of this article is the effort to produce a great chemical stable organic-inorganic hybrid nanocomposite with a great affinity to Pb(II), Cs(I), Cu(II), Sr(II), & Cd(II) cations from liquid solutions. Polyacrylonitrile acrylamide nano-silica was fabricated γ -irradiation within 50 kGy with comprehensive characterization.

3.1 Characterization

3.1.1 FT-IR

IR spectrum of {P(AN-AM)-NS} nanocomposite (Fig. 1). This Figure shows that broadband was noticed at 3470-3300 cm⁻¹ due to H₂O (stretching vibration) and OH adsorbed on {P(AN-AM)-NS} [19]. Two bands were noticed at 3190 & 2940 cm⁻¹ due to N-H (stretching vibration) & CH₂, respectively, [20–22]. A strong absorption band at 2244 cm⁻¹ due to the C≡N bond of AN (stretching vibration) [23]. 2 bands at 1661 & 1605 cm⁻¹, corresponding to amide I (stretching vibration) [24], and N-H of AM (bending vibration) or O-H bonded H₂O absorbed on {P(AN-AM)-NS} [20,25]. The band appeared at 1571 cm⁻¹ related to the C-N-H bond (bending vibration) [24]. 2 bands appeared at 1451 and 1413 cm⁻¹ related to an asymmetric & symmetric CH₂ (bending vibration) [20,26]. 2 bands at 1340 and

1220 cm^{-1} related to asymmetric & symmetric C-N (bending vibration) [24]. The bands appeared at 1040 & 905 cm^{-1} attributed to Si-O-Si & Si-O-H deformation vibration, respectively [27]. The bands at 636 & 460 cm^{-1} , attributed to Si-O [19].

3.1.2 XRD analysis

XRD analysis of {P(AN-AM)-NS} (Figure 2). It is clear that the prepared material has a crystalline structure and this result was parallel to the data acquired from XRD of {P(AM-AA)-MgSi} [9] and (KCFC-PAN) [28]. It can be established that the unlike indexed peaks at 30.6, 45.1, and 77.7° correspond to (031), (231), and (360) in {P(AN-AM)-NS} patterns (XRD JCPDS data file no. 43-1022) [9], a representative that it is supplemented with crystalline moieties. The motive for this is related to the presence of NS in {P(AN-AM)-NS}. Another peak looking at 20.9° corresponds to (020) is crossbanding to the occurrence of AM [9].

3.1.3 Thermal analysis

DTA&TGA chart of {P(AN-AM)-NS} (Fig. 3), via a three-steps. From 47 to 225 °C due to the reduction of surface moisture of polymeric resin [9], the weight loss is 10.5 %. From 225 to 505 °C due to the removal of H₂O of crystallization [3] and complete decay of the organic portion of the powder [29], the lost weight is 33.2 %. From 505 to 700 °C due to heating in N₂ consequences in a cyclization reaction with nitril bonds converted to C=N [29], the weight loss is 18.1 %. DTA displays that peak at 92 °C (endothermic) was attributed to the removal of surface moisture of polymeric resin. Two exothermic peaks appear at (353 and 533 °C), respectively, corresponding to the widespread decomposition of the organic fragment or due to heating in N₂ consequences in a cyclization reaction with nitril bonds converted to C=N. From TGA data (Fig. 3), the lost weight is continuous up to 600 °C. This supporting the fact that {P(AN-AM)-NS} has good thermal steadiness comparing with the different polymeric resins and the full lost weight with a heating temperature of 61.9 %.

3.1.4 Morphology structures (SEM)

Micrograph of {P(AN-AM)-NS} nanocomposite Figure (4A) showed an irregular, homogenous distribution of a large number of nanospheres spherical nanocomposite with the construction of a large number of cavities on the surface, which makes it easier to bring more active adsorption spots [22,23,30–32].

3.1.5 TEM

Transmission electron micrographs of {P(AN-AM)-NS} composite presented in Figure 4(B&C), exposed the presence of spherical units with sizes ranged from 2.3 to 4 nm. TEM images also showed no aggregations formation [33]. An experimentally noticed diffraction pattern viewing the central, intense, direct beam such a pattern, with a sharply intensive spot indicating that the specimen is at least moderately crystalline [34].

3.2 Chemical stability

The stability of {P(AN-AM)-NS} to chemical attack was studied in different solvents (H₂O, HNO₃, HCl, & KOH) and the results are tabulated in Table (1) and indicated that the prepared sample is stable in H₂O and (HNO₃ and HCl) up to 3 M, whereas the solid is moderately dissolved at 4 M HNO₃ and completely dissociated at 6 M, also the solid sample is stable in KOH up to 3 M, while partially dissociated at 4 M and fully dissociated at 6 M. The % solubility was raised by increasing of the acid & base concentrations [14]. Also, these results reflect that the sequence of stability of the studied composite is HCl > HNO₃ > KOH.

3.3 Sorption results

3.3.1 Distribution coefficients studies (K_d)

Fig. 5A, illustrates the variation of K_d of Pb²⁺, Cs⁺, Cu²⁺, Sr²⁺ and Cd²⁺ on {P(AN-AM)-NS}, as a function of pH. It is clear that the K_d rise by the increase in pH. The optimum K_d was achieved at pH 4.3 and was discovered to be (1655.6, 110.3, 91.7, 66.7, and 52.1 mL/g) for Pb(II), Cs(I), Cu(II), Cd(II), and Sr(II) ions, respectively. At a lower value (pH ≤ 3), K_d was inhibited, related to the fact that when the pH is quite little, the existence of excess H⁺ competes with Pb²⁺, Cs⁺, Cu²⁺, Sr²⁺ and Cd²⁺ in the liquor and preferably occupy the binding positions available in {P(AN-AM)-NS} [35,36]. At a higher value (pH > 3), K_d continuously rises with the increase in pH due to the reduction in H⁺ competition and maximum uptake carried out at pH=4.3 as well as uptake was slightly decreased at pH > 4.3, so all sorption experiments were done at pH 4.3. K_d and α_b^a for the mentioned cations at several pHs (2.4-6) were calculated and tabulated in Table 2, and designated that the K_d have the affinity arrangement; Pb²⁺ > Cs⁺ > Cu²⁺ > Cd²⁺ > Sr²⁺, this arrangement supported that the sorption of studied cations was applied in hydrated ionic radii. The cations with minor hydrated ionic radii simply enter the cavities of the polymeric resin resulting in higher sorption [12]. α_b^a were computed and designated that Pb²⁺ has very greater α_b^a by (31.8, 24.8, 18.1, and 15.0) for Sr²⁺, Cd²⁺, Cu²⁺, and Cs⁺ at top uptake (pH 4.3), these

values indicated that Pb(II) ions can simply separate from industrial and radioactive waste involved the above-mentioned cations.

Fig. 5B shows that the nonlinear relation between $\log k_d$ and pH was observed for studied cations. This relation reflects the non-reality of the exchange mechanisms. This variation may be due to taking place reactions other than ion exchange, similar precipitation, and/or adsorption [14].

3.3.2 Influence of shaking time

Shaking time impact on the removal efficiency of Pb^{2+} , Cu^{2+} , Sr^{2+} , Cs^+ , & Cd^{2+} on {P(AN-AM)-NS} was done at pH 4.3 and exposed in Fig. (6A). It can be understood that amount sorbed per unit gram of studied cations gradually increases with time and after 4 h the maximum removal of these ions was achieved and this time was used for further experimental work.

3.3.3 Kinetic investigation

The Lagergren pseudo-first-order (PFO) equation is stated as [2,13]:

$$\log(q_e - q_t) = -\left(\frac{K_f}{2.303}\right)t + \log q_e \quad (5)$$

In which K_f (min^{-1}) is a rate constant of PFO, q_e & q_t (mg/g) are the amount sorbed per gram at equilibrium & time t . Plotting $\log(q_e - q_t)$ beside t as exposed in Fig. (6B), the plot shows a linear relationship. The PFO suffered from lack when applied to Pb^{2+} , Cu^{2+} , Sr^{2+} , Cs^+ , and Cd^{2+} sorption on the composite sorbent. When the q_e achieved from Lagergren plots was associated with the q_e (experimental) considered one of the main discrepancies was observed. Thus, good linearity of Lagergren plots is not promised that the reaction of Pb^{2+} , Cu^{2+} , Sr^{2+} , Cs^+ , and Cd^{2+} with the sorbents don't follow PFO [37].

The pseudo-second-order (PSO) could be exemplified by the subsequent equation [38]:

$$\frac{t}{q_e} = \frac{1}{K_s q_e^2} + \frac{t}{q_e} \quad (6)$$

where K_s is PSO rate constant ($\text{g mg}^{-1} \text{ min}^{-1}$). Plots of t/q_t against t for Pb^{2+} , Cu^{2+} , Sr^{2+} , Cs^+ , & Cd^{2+} removal are exposed in Fig. (6C). A linear relationship is achieved and R^2 nearer to unity and clarifies that the sorption procedure surveys PSO kinetics. The initial sorption rate represented as $h = K_s(q_e)^2$, q_e , K_s , & R^2 were computed and arranged in Table (3). These results reveal that R^2 is nearer to unity for PSO than PFO. The q_e is consistent with the q_e (experimental). So, the sorption interaction can be approached more agreeably by the PSO. These data reveal that PSO is the chief interaction and K_s of Pb^{2+} , Cu^{2+} , Sr^{2+} , Cs^+ , and Cd^{2+} performs to be exact by the chemisorption method [38].

3.3.4 Capacity measurements and thermal stability

Heating temperatures affected the capacity of Pb^{2+} , Cu^{2+} , Sr^{2+} , Cs^+ , and Cd^{2+} on {P(AN-AM)-NS} in H^+ form was achieved, and the results were formulated in Table (4). The prominent reduction in the capacity was noticed with heating temperatures, corresponding to decay of the organic fragment of the solid as exposed in TGA&DTA data [5]. The capacity increases according to reduction in the hydrated ionic radii & hydration energy with sequence order; $\text{Pb}^{2+} > \text{Cs}^+ \gg \text{Cu}^{2+} > \text{Sr}^{2+} > \text{Cd}^{2+}$ [39,40]. The little capacity of this material for Cd^{2+} reflects the non-selectivity for this ion [14].

3.3.5 Thermodynamic studies

Fig. 7 displays the linear relation among $\ln K_d$ of Pb^{2+} , Cs^+ , Cu^{2+} , Sr^{2+} , and Cd^{2+} on {P(AN-AM)-NS} and $1000/T$ agreeing to the Van't Hoff equation [13];

$$\ln K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (7)$$

where (ΔS° , ΔH° , R , & T) are the change of entropy, the change of enthalpy, the gas constant, & absolute temperature, respectively. K_d of investigated cations improved with rising temperatures (298-338 K) (i.e., the K_d reduced with raising $1000/T$) as exposed in Fig. 7. This enhancement in sorption was corresponding to the quickening of some firstly slow adsorption phases and the creation of some fresh exchangeable sites on the sorbent layers [13]. From the slopes & intercepts displayed in Fig. 7, ΔH° & ΔS° were assessed and arranged in Table (5). +ve values of both ΔH° & ΔS° indicate the endothermic nature of the sorption process and bigger randomness of solid solution interface in the adsorption of these cations on {P(AN-AM)-NS}, respectively [13].

The change of free energy ΔG° was computed by the equation:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (8), \text{ and}$$

$$\Delta G^\circ = TR \ln K_d \quad (9)$$

-ve values of ΔG° represented in Table (5) reflect that the sorption is spontaneous and reveal the better sorption of these ions on {P(AN-AM)-NS} compared with H^+ ion [13].

4 Conclusion:

Polyacrylonitrile acrylamide nano-silica was fabricated, characterized, and employed for batch sorption of lead, cesium, copper, strontium, and cadmium ions from an liquid medium. {P(AN-AM)-NS} fabricated by γ -radiation initiated preparation at 50 kGy. The distribution coefficients at optimum pH have selectivity order: $\text{Pb}^{2+} > \text{Cs}^+ > \text{Cu}^{2+} > \text{Cd}^{2+} > \text{Sr}^{2+}$. The systems kinetic follow pseudo-second-order

kinetics. The capacity of studied cations reveals that {P(AN-AM)-NS} has affinity sequence; $\text{Pb}^{2+} > \text{Cs}^+ > \text{Cu}^{2+} > \text{Sr}^{2+} > \text{Cd}^{2+}$. Finally, thermodynamic parameters showed that the sorption interaction was spontaneous & endothermic.

Acknowledgments: This work has been supported by the Egyptian Atomic Energy Authority. Great thanks to all members of the nuclear fuel technology department, Egyptian Atomic Energy Authority for supporting this work.

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Figures

Fig. 1.

Figure 1

IR spectra for {P(AN-AM)-NS} nanocomposite.

Fig. 2.

Figure 2

XRD analysis for {P(AN-AM)-NS} nanocomposite.

Fig. 3.

Figure 3

TGA & DTA analysis for {P(AN-AM)-NS} nanocomposite.

Fig. 4.

Figure 4

(A) SEM micrograph (B) and (C) TEM images of {P(AN-AM)-NS} nanocomposite.

Fig. 5.

Figure 5

Sorption behavior of various metal ions onto {P(AN-AM)-NS} at 25 ± 1 °C, (A) Effect of pH on K_d (B) Plots of $\log K_d$ against pH.

Fig. 6.

Figure 6

Sorption of various metal ions on {P(AN-AM)-NS} at 25±1 °C (A) Effect of contact time on the amount sorbed, (B) Pseudo first-order kinetic plots, and (C) Pseudo second-order kinetic plots.

Fig. 7.

Figure 7

Van't Hoff plot of the adsorption of various metal ions on {P(AN-AM)-NS}.

Supplementary Files

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