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Synthesis and application of poly(Acrylamide-Itaconic Acid)/Zirconium tungstate composite material for cesium removal from different solutions

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ABSTRACT
This study is conducted to find the conditions required to synthesize composite material for cesium (\(^{134}\text{Cs}^+\)) removal from the generated liquid waste associated with nuclear, medical, industrial, and/or research activities. The study shows that the optimum conditions required for synthesizing “poly [acrylamide (AM)-itaconic acid (IA)]/N,N'-methyleneacrylamide (DAM)/zirconium tungstate (ZrW)” or “poly(AM-IA)/DAM/ZrW” are 0.01 g DAM dose as a cross-linker, a co-monomer concentration of 20%, a co-monomer composition (AM-IA) (12:88), and 0.03 g (melted at 450°C–500°C) ZrW with gamma irradiation dose of 30 kGy. The composite material was characterized by Fourier infrared (FTIR), X-ray diffraction (XRD), thermal gravimetric analysis (TGA), differential thermal analysis (DTA), scanning electron microscope (SEM), and Brunauer-Emmett-Teller (BET) surface area measurements. The adsorption performance of the composite was investigated. The maximum removal efficiency of \(^{134}\text{Cs}^+\) ions was found to be 93% in moderate alkaline solutions at pH 8 ± 0.2 after 90 min. Kinetic studies indicated that the adsorption process is controlled by the pseudo-second-order kinetic model as a chemisorption process. Fitting of the adsorption data has pointed out that the adsorption process follows the Freundlich isotherm model as heterogeneous process. The maximum adsorption capacity \((q_{\text{max}})\) is 5.298 mmol Cs\(^+\) g\(^{-1}\) adsorbent. Applicability of the synthesized composite material was also examined to remove \(^{134}\text{Cs}^+\) ions in different aqueous solutions.

KEYWORDS
\(^{134}\text{Cs}^+\); adsorption; isotherm; kinetic; liquid waste; polyamide composite; treatment

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Introduction
Radionuclides of \(^{134}\text{Cs}\) and \(^{137}\text{Cs}\) with the half-lives of 2.6 and 30 years, respectively, belong to the main long-lived fission products of \(^{235}\text{U}\). These radionuclides undergo radioactive decays by beta mode either to \(^{37}\text{Ba}\) or \(^{137m}\text{Ba}\) with \(\gamma\)-ray emission of 662 keV. One gram of \(^{137}\text{Cs}\) has an activity of 3.215 TBq. A considerable amount of \(^{137}\text{Cs}\) has been released into the environment over the years: ~10\(^{18}\) Bq in the Fukushima accident (Giani and Helmers 1997), and an estimated 10\(^{16}\) Bq in the Chernobyl accident (Yasunaria et al. 2011; Chino et al. 2011; Morino, Ohara, and Nishizawa 2011). In recent years, considerable interest has arisen with regard to the fate and transport of radionuclides such as \(^{137}\text{Cs}\) in aquatic environments. \(^{137}\text{Cs}\) is an important indicator of radioactive pollution in aquatic environments. The transport and fate of anthropogenic \(^{137}\text{Cs}\) are related to the chemical properties of ionic C\(_{s}^+\), which generally dictates a high degree of mobility and bioavailability of this radionuclide. Therefore, removal of the pollutants from industrial wastewaters has recently become one of the most important processes because of it becoming more profound with increasing industrial activities. Cesium in radioactive waste produced from industry, medicine, research and nuclear establishments is one of these pollutants that its separation from aqueous solution is mostly needed. In addition to some toxic organic compounds that are suspected to be carcinogens, these compounds can cause fatal damages (Fessenden, Fessenden, and Landgrebe 1986).

Studies on the removal of Cs\(^+\) ion from aqueous solution have been focused largely on adsorption and ion-exchange methods (El-Kamash 2008; Dahiya, Tripathi, and Hedge 2008; Chen and Wang 2008; Sangvanich et al. 2010; Younjin, Chorong, and Sang-June 2015; Abdel-Galil, Khalil, and El-Aryan 2015; Choong and Ju-Hyun 2015; Amin, Ali, and Mohammad 2015; Hun, Jung-Seok, and Wooyong 2015; Yang, Okada, and Nagatsu 2016). Natural and synthetic zeolites, clay minerals, and synthetic organic or inorganic ion exchangers have been used on a large scale for separation of radioactive Cs from low- and intermediate-level radioactive waste effluents or contaminated water (Panswed and Wongchaisuwan 1986; Smičiklas, Dimović, and Plečaš 2007; El-Kamash 2008; Sangvanich et al. 2010). However, the majority of these materials appear to be not economically attractive because of their high operational cost and poor performance. Also, many technologies have been developed for pollutant removal from aquatic environments, including flocculation and coagulation (Panswed and Wongchaisuwan 1986), membrane separation (Ciardelli, Cors, and Marucci 2000), oxidation or ozonation
Composite adsorbents/ion exchangers have been widely used for treatment of liquid wastes. The composite ion exchangers have improved qualities compared to pure organic or inorganic exchangers, such as better selectivity for the capture of some ions, increased mechanical and chemical resistance, more regularity form of the grains, smaller solubility in water, and better kinetics of exchange relative to the pure organic or inorganic exchangers. The composite ion exchangers are generally obtained by implantation of an inorganic part in the wide range of organic materials during the polymerization process (Kilincarslan and Akyil 2007). Linear low-density polyethylene-based and metal oxide (PbO and WO3)-filled composites were prepared and characterized for ionizing electromagnetic radiation (IEMR) shielding applications (Belgin and Aycik 2015). A graphene-based magnetic composite (Fe3O4/GO) was prepared, characterized, and utilized as an efficient adsorbent for immobilization of U(VI) from aqueous solutions in nuclear waste management (Donglin et al. 2015; Shujun et al. 2015). New composite of cellulose, chitin, and chitosan modified with Ti or Ni nanoparticles was developed and tested for removal of 134Cs, 85Sr, 60Co, and 152+154Eu from radioactive wastewater (Jana et al. 2015). Graphene oxide (GO) and two magnetic graphene oxide (MGO) composites with a different amount of magnetite were synthesized and used for sorption of Am3+ and Pu4+ isotopes as well as Co2+, Ni2+, Cu2+, and Pb2+ (Galina et al. 2015). The titrinate/GO composites were successfully synthesized by thermal treatment and applied as a new adsorbent for removal or preconcentration of radiocobalt (60Co) and other kinds of radionuclides from aqueous solutions in environmental pollution cleanup (Xiangxue, Songsheng, and Mancheng 2015). A novel composite material of SiO2–Al2O3 based on poly(acrylic acid–maleic acid) was synthesized, characterized, and used for removal of 134Cs in different concentrations of acid or salt solutions. The poly(AA–MA)/SiO2/Al2O3 composite is a promising adsorbent for Cs removal from acidic liquid radioactive waste (Attallah, Allan, and Mahmoud 2015).

The novelty statement of the present work is in optimizing the conditions required to synthesize organic/inorganic composite, namely “poly(AM-IA)/DAM/ZrW.” Herein, the composite material was characterized and applied as an efficient adsorbent for removal of 134Cs+ radionuclide from different aqueous solutions. The sorption performance of 134Cs+ ions onto the composite surface was also investigated by different kinetic and isotherm models.

**Materials and methods**

**Reagents**

All chemical reagents were of analytical grade (AR grade). Distilled water was used for preparing the working solutions. The reagents of acrylamide (AM, 98%), itaconic acid (IA, 99%), and N,N’-methyleneacrylamide (DAM) were provided by Merck (Germany). Zirconium hydrous oxide (99%) was obtained from Riedel (Germany). Sodium tungstate (98%) was obtained from LOBA Chemie Company.

**Preparation of the composite material**

**Preparation of zirconium tungstate (ZrW)**

The inorganic ZrW was prepared by mixing equimolar (0.1 M) and equal volumes (250 mL) of zirconyl chloride (ZrOCl2·8H2O) and sodium tungstate (Na2WO4·2H2O) solutions with constant stirring at ambient temperature (Wu, Badarinayanan, and Kessler 2012). The pH of the reaction mixture was adjusted (pH 8) and stirred for 60 min. The precipitate was filtered and washed with distilled water to constant pH 4.5 and dried at 50°C for 24 h. The produced granulated white material was grounded and sieved. The fraction below 0.25 mm was taken for further work.

**Preparation of co-monomer solution**

The AM and IA were dissolved in ethanol mixture (1:1 v/v), and the crosslinker DAM was dissolved in deoxygenated water at the required concentrations to form (AM-IA)–DAM mixture.

**Preparation of composite material**

The proposed organic–inorganic polymer composite material was prepared by the direct mixing of the inorganic component with the polymer matrix. Mixing can be achieved by either solution or melt blending, provided both components may be dispersed in a common solvent or melt at high temperature. Melt blending is more common because of its efficiency and environmental containment (Zhang, Rong, and Friedrich 2003). After the solution was irradiated in 60Co γ-ray source, a white viscous product was obtained in acidic medium (pH 3) which was then washed with acetone to remove the free monomers. Finally, the product of the synthesized composite material was dried at 80°C for 24 h.

**Radioactive isotope**

For the radiochemical investigations, radioactive isotope of 134Cs was produced through the irradiation of cesium carbonate at the Egyptian second Research Reactor (ETRR-2). Accurate amounts of cesium carbonate (Cs2CO3) samples (10 mg) were wrapped in thin aluminum foils that were previously cleaned with acetone and finally placed in thick aluminum irradiation capsules. They were transferred to an aluminum irradiation box of 670 mm length and then irradiated in the ETRR-2 core adjacent to thermal neutron flux of 1013 n. cm−2.s−1 for about 24 h.

**Batch uptake studies**

The initial pH of the solutions was adjusted by adding either HCl or NaOH solution. The adsorption behavior of the prepared composite material as a function of pH and different media has been performed using batch method. For this purpose, 10 mL of the aqueous solutions of radioactive 134Cs+ was shaken with 0.05 g of the composite material poly (AM-IA)/DAM/ZrW at 25°C ± 1°C. Thereafter, 2 mL from...
Cs\(^+\) solution was withdrawn for the radiometric measurement. The uptake (A, %) on the solid could be calculated by Equation (1) (El Afifi, Attallah, and Borai 2016):

\[
A = \left( \frac{C_i - C_f}{C_i} \right) \times 100
\]

where \(C_i\) and \(C_f\) are the initial and final net counts of \(^{134}\text{Cs}\), respectively.

Sorption isotherm experiments were conducted batch wise. In this concern, the equilibrium was attained by shaking 0.05 g of the poly(AM-IA)/DAM/ZrW composite in 10 mL of aqueous solutions containing 0.004–400 mmol · L\(^{-1}\) of Cs\(^+\) and tracer of the radioactive isotope (\(^{134}\text{Cs}\)) at constant pH, temperature (°C), and contact time (min). The initial and equilibrium concentrations of metal ion in the aqueous phase were determined, and the amount of metal ion per gram (\(q\), mmol · g\(^{-1}\)) of the composite material was obtained using Equation (2) (El Afifi, Attallah and Borai 2016):

\[
q = \left( \frac{A_i - A_f}{A_i} \right) C_0 \frac{V}{m}
\]

where \(A_i\) and \(A_f\) are the initial and the final activity of \(^{134}\text{Cs}\) and \(C_0\) is the initial concentration of Cs-carrier (mmol · L\(^{-1}\)). \(V\) and \(m\) are the volume of the sample solution (Liter) and the weight (g) of composite material, respectively.

**Results and discussion**

**Optimizing parameters of the prepared composite**

Table 1 summarizes the different parameters affecting the preparation of the prepared composite. The \(\gamma\)-radiation induced copolymerization of AM with IA in the presence of DAM as a crosslinker and zirconium tungstate (ZrW) as an inorganic material was studied by a free radical mechanism to determine the optimum conditions to produce an efficient poly(AM-IA)/DAM/ZrW as an exchanger. The effects of (i) DAM, (ii) comonomer concentration, (iii) comonomer composition, (iv) amount of melt inorganic zirconium tungstate (ZrW), and (v) radiation dose on the adsorption capacity toward Cu\(^{2+}\) were studied. From the experimental data (Table 1), it can be seen that the optimum conditions for preparing the composite material after studying the above parameters are 0.01 g DAM as a crosslinker, a comonomer concentration 20%, a comonomer composition (AM:IA) (12:88), and 0.03 g ZrW with radiation dose 30 kGy. The polymerization is achieved by free radicals (occasionally ions) created in the material at the end of the process. When AM and IA are irradiated with ionizing radiation by \(\gamma\)-rays in aqueous solutions, free radicals are generated. Random reactions of these radicals with the monomers lead to the formation of copolymers of AM/IA.

**Characterization of the Composite Material**

Figure 1 illustrates the FTIR spectra of the free poly(AM-IA)/DAM/ZrW composite (a) and after adsorption of Cs\(^+\) ions (b). It is observed that the broad absorption peak at 3446 cm\(^{-1}\) is

<table>
<thead>
<tr>
<th>Experimental condition</th>
<th>DAM dose</th>
<th>[Co-monomer], %</th>
<th>Co-monomer compositions</th>
<th>ZrW, g</th>
<th>Dose, kGy</th>
<th>Capacity, (q_e) (mmol · g(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>i. DAM concentration</td>
<td>0.01</td>
<td>10%</td>
<td>9:0:10</td>
<td>0.03</td>
<td>20</td>
<td>1.17</td>
</tr>
<tr>
<td></td>
<td>0.03</td>
<td></td>
<td>10:90</td>
<td>0.03</td>
<td>20</td>
<td>1.84</td>
</tr>
<tr>
<td></td>
<td>0.05</td>
<td></td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>1.91</td>
</tr>
<tr>
<td></td>
<td>0.07</td>
<td></td>
<td>16:84</td>
<td>0.03</td>
<td>20</td>
<td>1.82</td>
</tr>
<tr>
<td>ii. Co-monomer composition (AM:IA)</td>
<td>0.01</td>
<td>10%</td>
<td>2.98</td>
<td>0.03</td>
<td>20</td>
<td>0.316</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>8.92</td>
<td>0.03</td>
<td>20</td>
<td>0.911</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>1.18</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>16:84</td>
<td>0.03</td>
<td>20</td>
<td>1.78</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>20:80</td>
<td>0.03</td>
<td>20</td>
<td>2.73</td>
</tr>
<tr>
<td>iii. [Co-monomer], %</td>
<td>0.01</td>
<td>5%</td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>1.18</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>10%</td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>1.84</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>15%</td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>2.15</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>2.73</td>
</tr>
<tr>
<td>iv. Added ZrW, g</td>
<td>0.01</td>
<td>20%</td>
<td>12:88</td>
<td>0.03</td>
<td>20</td>
<td>1.17</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>30</td>
<td>0.03</td>
<td>20</td>
<td>2.55</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>40</td>
<td>0.03</td>
<td>20</td>
<td>3.22</td>
</tr>
<tr>
<td></td>
<td>0.01</td>
<td>20%</td>
<td>50</td>
<td>0.03</td>
<td>20</td>
<td>3.95</td>
</tr>
</tbody>
</table>

The optimized value for each parameter is bolded.
attributed to the stretching vibration of the bonded OH groups of the water molecules having intermolecular H-bonds and a peak at 1650 cm\(^{-1}\) which can also be attributed to water molecules.

As a result of H-O-H, the hydrogen ions can easily move on the surface of ZrW. So it acts as cation exchanger (Galsden 1975). The bands observed at 1454 cm\(^{-1}\) correspond to symmetric stretching vibrations of carboxyl groups, while the bands at 1315 cm\(^{-1}\) correspond to \(-\text{COO}^–\) bending group (Allan, Siyam, and Sanad 2001). The difference in both spectra can be due to the possible chelating between the active sites, e.g., carboxylate in the composite surface and Cs\(^+\) ions. This was confirmed by the significant difference in the band which appeared at 1650 cm\(^{-1}\). The interaction between the active sites of the synthesized composite with the metal ion, e.g., Cs\(^+\), was also ensured by the appearance of a band at \(~700\) cm\(^{-1}\) which may be due to the metal oxygen bonds (M \(-\text{O}\) bonds (Socrates 1980; Boukhalfa 2010).

The XRD spectrum of the prepared composite material showed a large broadening of the diffraction maxima. These broadenings can be attributed to the disordering character of the synthesized composite. Consequently, the composite can be considered as an amorphous adsorbent in nature.

The TGA and DTA of the composite poly(AM-IA)-DAM/ZrW was done with a heating rate of 10 °C.min\(^{-1}\) as given in Figure 2. It is observed that the behavior of TGA occurred via main four-stage processes (Lanthong, Nuisin, and Kiatkamjornwong 2006). The first stage was within 75 °C–171 °C. This may be attributed to the removal of humidity attached to surface and/or matrix from the polymeric resin with weight loss \(~7\)%. The second stage occurred between 171 °C and 300 °C due to the decarboxylation of IA and removal of CO\(_2\) gas. The weight loss of this stage was \(~17\)%.

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The third stage was at 300 °C–540 °C due to the degradation of the AM portion with removal of NH\(_3\) gas and weight loss percentage of \(~40.5\)%.

The last stage was from 540 °C to 800 °C due to chain scission and removal of the volatile hydrocarbons with weight loss \(~27.4\)%.

Beyond 800 °C, there is complete decomposition to the inorganic part.

The DTA displayed many endothermic peaks. The first one is at 97.3 °C, due to the removal of the external water molecules. Two endothermic peaks at 198.2 °C and 242.9 °C are attributed
to decarboxylation of IA and removal of CO₂ gas. Two endothermic peaks at 409 °C and 507 °C can be due to the release of ammonia gas. On the other hand, there is an exothermic peak at 715 °C corresponding to the chain scission and finally the formation of the metal oxides and/or carbide.

The emission scanning electron microscopy (SEM) was used to observe the surface morphology of the prepared composite poly(AM-IA)/DMA/ZrW material before and after loading with Cs⁺ ions. Figure 3 (a and b) illustrates the SEM micrographs of the prepared composite and after adsorption of Cs⁺ ions. From these photomicrographs, it can be seen that the porous structure of the synthesized material has a porous basic structure, Figure 3a. Thereby, the material of this composite can be used as an adsorbent. It could be observed that there is a distinct change in the surface morphology after adsorption. Figure 3b illustrates the SEM micrographs of composite by the loading with Cs⁺ ions. Therefore, the clear difference between both images (a and b) supports that the synthesized composite material has a reactive site and affinity toward the metal ions (e.g., Cs⁺).

Furthermore, the surface analysis of the prepared composite has shown that the surface area and the interior pore volume are 15.4 m² g⁻¹ and 1.92 × 10⁻² cc · g⁻¹, respectively. Thereby, the prepared poly(AM-IA)/DMA/ZrW composite can be used as a suitable adsorbent material for removal and/or immobilization of ¹³⁴Cs⁺ in the next sub-sections.

**Sorption performance of ¹³⁴Cs⁺**

The poly(AM-IA)/DMA/ZrW composite contains carboxylate (–CO₂⁻) and amide groups (–CO.NH⁻) which are of anionic type superabsorbent polymers. It is well known that this type normally ionizes at a high pH but unionizes at a low pH. The first and second dissociation constants of the IA in the composite are pKa₁ ≈ 3.85 and pKa₂ = 5.44, respectively, as described by Sen et al. (1999).

Under acidic conditions (pH ≤ 6), the carboxylate groups are protonated and the copolymer network shrinks significantly. If the composite material is exposed to an alkaline medium at pH ~ 8 for a longer time, it can hydrolyze AM moiety to acrylic acid moiety, which is also an anionic type superabsorbent. Thus, it can be concluded that the adsorption uptake of Cs⁺ ions is high in alkaline media till pH 8 followed by a gradual decrease. Therefore, the adsorption reaction of Cs ions onto composite can proceed according to Equation (3):

\[
\text{O} \quad \text{R} \quad \text{O}
\]

\[
\text{\underbrace{-\text{C-OH}}_{\text{carboxylic groups on composite surface}}} + n\text{Cs}^+ \quad \text{pH} > 8 \quad \text{\underbrace{-\text{C-O}}_{\text{carboxylic groups on composite surface}}} + n\text{H}^+ \quad (3)
\]

where \( R = \text{composite pattern} \) (poly(AM-IA)/DMA/ZrW), \( -\text{COOH} = \text{carboxylic groups on composite surface} \), \( n\text{H}^+ = \text{number of protons released} \). Figure 4 demonstrates the influence of the hydrogen ion concentration (i.e., pH) on the adsorption performance of radiotracer ¹³⁴Cs⁺ ions from aqueous solution within pH 1.5–12 by using the synthesized material.

*Figure 2.* The thermal analysis (TGA and DTA) of the synthesized composite material.

*Figure 3.* The SEM micrograph of (a) free composite and (b) composite loaded by Cs⁺.
composite material. The maximum retention efficiency (%) of radiotracer $^{134}$Cs onto surface of the composite reached $\sim 93\%$ in moderately alkaline medium at pH 8 ± 0.2.

**Adsorption kinetics**

The kinetics of adsorption process of Cs$^+$ ions onto the synthesized composite poly(AM-IA)/DAM/ZrW was carried out at constant temperature 25°C ± 1°C, V/m ratio 0.2 l.g$^{-1}$, pH 8, and metal ion concentration of 0.04 mM. The adsorption of Cs$^+$ on the adsorbent material has been investigated as a function of contact time (5–120 min) and the data obtained are illustrated in Figure 5.

It was found that adsorption of Cs$^+$ increased gradually during the first 1 h of the adsorption process, saturated slowly, and reached a maximum adsorption capacity at the time of equilibrium after 90 min. The highest retention efficiency ($R$, %) and uptake ($q_t$) of $^{134}$Cs$^+$ onto the synthesized composite material were $\sim 93\%$ and 0.0074 mmol · g$^{-1}$, respectively. Therefore, 90 min was chosen as enough contact time between Cs ions and the adsorbent to obtain the maximum adsorption capacity in the further investigations.

On the other hand, the adsorption data were studied according to three kinetic models to verify the order of adsorption performance. The pseudo-first-order kinetics model was tested according to Equation (4) (Doniaa et al. 2009; Feihong et al. 2015; Ümit et al. 2015, El Afifi, Attallah, and Borai 2016):

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t$$

where $q_e$ and $q_t$ (mmol · g$^{-1}$) refer to the amount of Cs$^+$ ions adsorbed at equilibrium and at time (t), respectively; $k_1$ is the rate constant of pseudo-first-order (min$^{-1}$). Values of $k_1$ and $q_e$ were calculated from the slope and intercept values of the straight line of log ($q_e - q_t$) versus (t); the results are shown in Figure 6 and reported in Table 2.

The adsorption data were also treated according to the pseudo-second-order kinetics using Equation (5) (Yu et al. 2013):

$$\frac{t}{q_t} = \frac{1}{k_2q_e^2} + \frac{1}{q_e} \frac{1}{t}$$

where $k_2$ is the overall rate constant of the pseudo-second-order adsorption. The plot of $t/q_t$ versus (t) gives a straight line with slope and intercept equal to 1/$q_e$ and 1/$k_2q_e^2$, respectively. The calculated values of $q_e$ and $k_2$ were obtained from Figure 7 and are reported in Table 2.

An intraparticle mass transfer diffusion model proposed by Weber and Morris can be written as given in Equation (6) (Machida, Aikawa, and Tatsumoto 2005):

$$q_t = k_{id}t^{1/2} + C$$

where $C$ (mmol · g$^{-1}$) is the intercept and $K_{id}$ (mmol · g$^{-1}$ · min$^{-1/2}$) is the intraparticle diffusion rate constant which can be calculated from the slope of the linear plots of $q_t$ versus $t^{1/2}$. The calculated values of $c$ and $K_{id}$ were obtained from Figure 8 and are reported in Table 2.

One of the major discrepancies was observed when $q_e$ values obtained from straight line of plotting were compared with the experimental sorption capacity ($q_e$) value (Table 2).
The experimental $q_e$ value (0.0074 mmol·g$^{-1}$) differs from the corresponding calculated value (0.0060 mmol·g$^{-1}$). Thus, the good linearity of the plot does not guarantee that the interaction of Cs$^+$ with the synthesized sorbent will follow first-order kinetics. On the other hand, the calculated $q_e$ based on the data fitted by the pseudo-second-order kinetic model is closer (0.0070 mmol·g$^{-1}$) to experimental value as given in Table 2. Therefore, the sorption reaction can be approximated more favorably by the pseudo-second-order kinetic model. These results suggest that a pseudo-second-order sorption is the predominant mechanism, and the overall rate constant of Cs$^+$ ions appears to be controlled by the chemisorption process. The rate of adsorption process is less controlled by the intraparticle mass transfer diffusion mechanism due to the moderate bulky size of Cs$^+$ ions. This implies that the adsorption of Cs$^+$ is dependent on the concentration of both metal ions and active sites of the composite characters.

### Adsorption isotherm

The study of the concentration dependency was done by changing the initial concentration of Cs$^+$ ions in the range 0.004–400 mmol·L$^{-1}$ at 25°C, pH 8, and contact time 90 min. The results plotted in terms of the amount adsorbed ($q_e$, mmol·g$^{-1}$) versus the equilibrium concentration of Cs$^+$ ions in the aqueous phase ($C_e$, mmol·L$^{-1}$) onto the prepared composite poly(AM-IA)/DAM/ZrW are demonstrated in Figure 9.

It was found that the amount adsorbed increased as the equilibrium concentration of the ions increased, with a nearly constant value being achieved at higher equilibrium concentrations. This is a common phenomenon in adsorption processes which is brought about by the gradual saturation of adsorption sites on the adsorbent surface so that a further addition of ions to the aqueous phase surrounding the adsorbent particles would not be expected to increase the amount adsorbed significantly (Machida, Aikawa, and Tatsumoto 2005; Doniaa et al. 2009; Yu et al. 2013, Feihong et al. 2015; Ümit et al. 2015; El Afifi, Attallah, and Borai 2016). In this study, the maximum capacity ($q_e$) of Cs$^+$ onto the composite surface reached 5 mmol·g$^{-1}$ that corresponds to equilibrium concentration ($C_e$) of 175 mmol·L$^{-1}$. The further increase in equilibrium concentration is associated with gradual decrease in the adsorption capacity of Cs$^+$.

### Table 2. The kinetic parameters of $^{134}$Cs$^+$ adsorbed onto poly(AM-IA)/DAM/ZrW composite.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experimental maximum uptake, $q_e$ (mmol·g$^{-1}$)</td>
<td>0.0074</td>
</tr>
<tr>
<td>$q_e$ calculated (mmol·g$^{-1}$)</td>
<td>0.0070</td>
</tr>
<tr>
<td>$k_2$ (g·mmol$^{-1}$·min$^{-1}$)</td>
<td>11.915</td>
</tr>
<tr>
<td>$R^2$</td>
<td>0.94</td>
</tr>
<tr>
<td>$k_{id}$ (mmol·g$^{-1}$·min$^{-1}$)</td>
<td>0.0029</td>
</tr>
<tr>
<td>$c$ (g·mmol$^{-1}$·min$^{-1}$)</td>
<td>0.00016</td>
</tr>
<tr>
<td>$R^2$</td>
<td>0.88</td>
</tr>
</tbody>
</table>

$^c$ calculated.

![Figure 7](image-url) The pseudo-second-order plots for adsorption of $^{134}$Cs$^+$ ions onto synthesized poly(AM-IA)/DAM/ZrW composite.

![Figure 8](image-url) The Weber–Morris plots for adsorption of $^{134}$Cs$^+$ ions onto synthesized poly(AM-IA)/DAM/ZrW composite.

![Figure 9](image-url) The relation between uptakes ($q_e$) of $^{134}$Cs$^+$ ions onto synthesized poly(AM-IA)/DAM/ZrW composite and equilibrium concentration ($C_e$).
The results of adsorption equilibrium isotherm were evaluated and described by using three isotherms: Langmuir, Freundlich, and Temkin adsorption isotherm equations (Ümit et al. 2015; El Afifi, Attallah, and Borai 2016). The most widely used isotherm equations for modeling equilibrium data is the Langmuir model. It is represented by Equation (7) which assumes that the adsorbent surface is homogeneous and the adsorption sites are energetically identical, indicating that the adsorbed molecules do not react with each other. The linear form of Langmuir equation is given by the equation (Feihong et al. 2015; El Afifi et al. 2016):

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{K_L q_{max}}$$  

where $C_e$ represents the equilibrium concentration of ions in solution (mmol · L$^{-1}$), $q_e$ refers to the amount adsorbed onto the adsorbent material (mmol · g$^{-1}$), $q_{max}$ is the maximum adsorption capacity (mmol · g$^{-1}$), and $K_L$ is the binding constant which is related to the energy of adsorption (L · mol$^{-1}$g$^{-1}$). Plotting $C_e/q_e$ against $C_e$ gives a straight line with slope and intercept equal to $1/q_{max}$ and $1/K_L q_{max}$, respectively (Figure 10). The values of $K_L$ and $q_{max}$ are reported in Table 3.

The values of $K_L$ and $q_{max}$ are 0.042 L · mmol$^{-1}$ and 5.298 mmol · g$^{-1}$, respectively. The degree of suitability of the synthesized composite material toward metal ions was estimated from the values of the separation factor constant ($R_L$) which gives an indication of the possibility of the adsorption process to proceed, with $R_L > 1$ being unsuitable; $R_L = 1$ linear; $0 < R_L < 1$ suitable; and $R_L = 0$ irreversible (Attallah, Allan, and Mahmoud 2015; El Afifi, Attallah, and Borai 2016). The value of $R_L$ could be calculated from Equation (8):

$$R_L = \frac{1}{1 + K_L C_o}$$  

where $C_o$ (mmol · L$^{-1}$) is the initial concentration of Cs$^+$ ions. The $R_L$ value ranges from 0.0375 to 0.9998 between 300 and 0.004 mmol · L$^{-1}$ and approaches zero with an increase in the $C_o$ value. This value ($0 < R_L < 1$) indicates the suitability of the synthesized poly(AM-IA)/DAM/ZrW composite to be used as adsorbent material for Cs$^+$ removal from radioactive aqueous solution.

The Freundlich isotherm model (Feihong et al. 2015; Ümit et al. 2015) is an empirical relationship describing the sorption of solutes from a liquid to a solid surface and assumes that different sites with several sorption energies are involved (the surface of the adsorbent is heterogeneous). The linear form of the Freundlich isotherm model can be represented by Equation (9) (Dada et al. 2012; Attallah, Allan, and Mahmoud 2015):

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$  

where $K_F$ (mmol · g$^{-1}$) and $n$ are characteristic constants related to the relative sorption capacity of the sorbent and the intensity of sorption, respectively. Plotting $\log q_e$ against $\log C_e$ gives a straight line with slope and intercept equal to $1/n$ and $\log K_F$, respectively. The parameter $n$ (i.e., 1.80) characterizes the heterogeneity of the system. Figure 11 shows the adsorption isotherm of Cs$^+$ ions, which conforms to the Freundlich equation.

The Freundlich plot gave a slope less than 1, indicating nonlinear sorption behavior with the concentration of Cs$^+$ in the range studied. The observed value of $K_F$ of Cs$^+$ onto the prepared composite material was found to be 0.24 mmol · g$^{-1}$ and confirms a significant affinity of the Cs ions to the studied composite.

The data were also represented by the Temkin absorption isotherm model. This model contains a factor that explicitly takes into account the adsorbent–adsorbate interactions. The linearized form of the Temkin isotherm model is described by Equation (10) (Dada et al. 2012; Feihong et al. 2015; Ümit et al. 2015; El Afifi, Attallah, and Borai 2016):

$$q_e = \frac{RT}{b_T} \ln k_T + \frac{RT}{b_T} \ln C_e$$  

where $b_T$ is the Temkin constant related to the heat of sorption (kJ · mol$^{-1}$), $k_T$ is the equilibrium binding constant corresponding to the maximum binding energy (L · g$^{-1}$), $T$ is the absolute temperature (K), and $R$ is the gas constant (8.314 J · mol$^{-1}$ · K$^{-1}$). The values of $k_T$ and $b_T$ were obtained from the slope and intercept of a straight line plot of $q_e$ versus $\ln C_e$ (Figure 12), respectively, and their values are given in Table 3.

It can be seen that the value of correlation coefficient ($R^2$) of Freundlich (0.99) equation is higher than that found for Langmuir (0.95) or Temkin (0.63) isotherm model. Thereby, the Freundlich isotherm correctly fits the equilibrium data with high correlation near unity and equals to 0.99. This supports that the adsorption of Cs$^+$ was preceded by means of different sites, e.g., carboxylate and/or amide moieties with several sorption energies. Therefore, the sorption reaction can be more favorable by Freundlich model, confirming the heterogeneous chemisorption of Cs$^+$ onto the composite surface.

To evaluate the adsorption efficiency of the synthesized composite, we compared our results with those found in literature (Attallah, Allan, and Mahmoud 2015; Allan, Holiel, and Sanad 2014; Hamoud et al. 2014; El-Gammal et al. 2009; Zhang...
demonstrated in Figure 14. It was observed that the removal of Cs ions from organic acid solutions was also studied and the efficiency reached 40\% depends on the hydrogen ion concentration. The removal efficiency varied with simulated solutions of Cs\textsuperscript{+} ions in the presence of three organic acids including acetic, citric, and ascorbic acids. The removal percentage was higher at low concentrations and decreased with the increase in their concentrations. Thereby, the sequence of the removal efficiency can be ordered at \([C] = 10^{-5}\) M as citric (42\%) > acetic (33\%) > ascorbic (28\%), whereas at \([C] = 10^{-1}\) M the sequence is citric (24\%) > acetic (13\%) > ascorbic (4\%).

Figure 15 shows the removal percentage of Cs\textsuperscript{+} ions from solution salts such as NH\textsubscript{4}Cl, di-sodium of EDTA and NaCl by the composite. It was found that the removal percentage of Cs\textsuperscript{+} ions increased with the decrease in solution concentration used. Thereby, the removal percentage of Cs\textsuperscript{+} is 1.3\%, 3.6\% and 9\% at 0.1 M NH\textsubscript{4}Cl, Na\textsubscript{2}EDTA, and NaCl, respectively. With the decrease in concentration to below \(10^{-5}\) M, the removal percentage of Cs\textsuperscript{+} increased to \(\sim 27\%\), 31\%, and 45\% from NH\textsubscript{4}Cl, Na\textsubscript{2}EDTA, and NaCl solutions, respectively. To learn more about the sorption behavior of the prepared composite, it was used to remove Cs\textsuperscript{+} ions from an aqueous solution containing organic solvent such as acetone. The results obtained are shown in Figure 16. It is noticed that the removal efficiency of Cs\textsuperscript{+} increases with the concentration of acetone till 60\% (v/v). The maximum removal obtained was above 60\% of Cs\textsuperscript{+} ions. Based on the aforementioned results, the lowest removal efficiency of Cs\textsuperscript{+} at high concentrations could be due to the competitive ions such as H\textsuperscript{+} or NH\textsubscript{4}\textsuperscript{+} or Na\textsuperscript{+} onto the prepared composite surface. Whereas the high removal efficiency of Cs\textsuperscript{+} from diluted solutions and/or at low concentrations may be attributed to the decrease in the competition of ions and/or the increase in the ionization rate of the active sites in surface of the prepared poly(AM-IA)/DAM/ZrW composite.

Table 3. The parameters for the Langmuir, Freundlich, and Temkin models of \(^{134}\text{Cs}^+\) sorption by the synthesized poly(AM-IA)/DAM/ZrW composite.

<table>
<thead>
<tr>
<th>Langmuir constants</th>
<th>Freundlich constants</th>
<th>Temkin constants</th>
</tr>
</thead>
<tbody>
<tr>
<td>$q_{\text{max}}$, mmol·g(^{-1})</td>
<td>$K_L$, L·mmol(^{-1})</td>
<td>$R^2$</td>
</tr>
<tr>
<td>5.298</td>
<td>0.042</td>
<td>0.95</td>
</tr>
</tbody>
</table>

Xu, and Yan 2010; Miah et al. 2010; El-Naggar et al. 2012; Omar and Attia 2013; Kim, Lee, and Jeon 2014; Khotimchenko et al. 2014). Table 4 shows the maximum adsorption capacity ($q_{\text{max}}$) for various adsorbent materials for adsorption of Cs\textsuperscript{+}, although a direct comparison of the adsorption capacity between the synthesized poly(AM-IA)/DAM/ZrW composite used in this study and other adsorbents reported in literature was difficult due to the variation with experimental conditions applied in those studies.

**Applicability of the prepared composite**

This part describes the possible applicability of the synthesized poly(AM-IA)/DAM/ZrW composite in the removal of Cs\textsuperscript{+} ions and/or its radioactive isotope such as \(^{134}\text{Cs}^+\) or \(^{137}\text{Cs}^+\) from the wastes produced from the industrial, medical, or research centers. Therefore, our investigations were carried out to remove Cs from different media in a wide range of concentrations ranging from \(10^{-1}\) to \(10^{-5}\) M, considering the optimum conditions obtained (pH 8, V/m ratio 0.2 L.g\(^{-1}\), contact time 90 min, free tracer of \(^{134}\text{Cs}^+\)). Figure 13 represents the use of the composite in the removal of Cs\textsuperscript{+} ions which exist in wastes containing acidic solutions of mineral acids such as HCl or HNO\textsubscript{3}. It was found that the removal efficiency (%) depends on the hydrogen ion concentration. The removal efficiency reached 40\% and 34\% of Cs\textsuperscript{+} ions from HCl and HNO\textsubscript{3} solutions at \(10^{-5}\) M, while it decreased to 6.5\% and 13\% in \(10^{-1}\) M.

The use of the composite as adsorbent for removal of Cs ions from organic acid solutions was also studied and demonstrated in Figure 14. It was observed that the removal efficiency varied with simulated solutions of Cs\textsuperscript{+} ions in the presence of three organic acids including acetic, citric, and ascorbic acids. The removal percentage was higher at low concentrations and decreased with the increase in their concentrations. Therefore, our investigations were carried out to remove Cs from different media in a wide range of concentrations ranging from \(10^{-1}\) to \(10^{-5}\) M, considering the optimum conditions obtained (pH 8, V/m ratio 0.2 L.g\(^{-1}\), contact time 90 min, free tracer of \(^{134}\text{Cs}^+\)). Figure 13 represents the use of the composite in the removal of Cs\textsuperscript{+} ions which exist in wastes containing acidic solutions of mineral acids such as HCl or HNO\textsubscript{3}. It was found that the removal efficiency (%) depends on the hydrogen ion concentration. The removal efficiency reached 40\% and 34\% of Cs\textsuperscript{+} ions from HCl and HNO\textsubscript{3} solutions at \(10^{-5}\) M, while it decreased to 6.5\% and 13\% in \(10^{-1}\) M.

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**Figure 11.** Freundlich plots for adsorption of \(^{134}\text{Cs}^+\) ions onto synthesized poly(AM-IA)/DAM/ZrW composite.

**Figure 12.** Temkin plots for adsorption of \(^{134}\text{Cs}^+\) onto synthesized poly(AM-IA)/DAM/ZrW composite.
In this work, poly(AM-IA)/DAM/ZrW composite material was synthesized and characterized by different measurements (FTIR, XRD, TGA, DTA, SEM, and BET). It summarizes that: (i) the optimum conditions for preparing the composite material are 0.01 g DAM as a crosslinker, a co-monomer concentration 20%, a co-monomer composition (AM-IA) (12:88), and 0.03 g ZrW with gamma irradiation dose 30 kGy; (ii) the maximum adsorption performance of the composite is 93% for radiotracer and ions of $^{134}$Cs$^+$ in aqueous solution at pH 8 after 90 min; (iii) sorption process of Cs$^+$ ions was favored by the pseudo-second-order kinetic model and controlled by the Freundlich equation model, confirming the heterogeneous chemisorption process with adsorption capacity ($q_{\text{max}}$) of 5.29 mmol·g$^{-1}$; and (iv) applicability of the adsorbent was also examined to remove Cs$^+$ ions or its radioactive isotopes $^{134}$Cs$^+$ and/or $^{137}$Cs$^+$ from different solutions. It is interesting to

### Table 4. Comparing the adsorption capacity ($q_{\text{max}}$) of poly(AM–IA)/DAM/ZrW for cesium with those reported in literatures using other materials.

<table>
<thead>
<tr>
<th>Adsorbent material</th>
<th>pH media</th>
<th>$q_{\text{max}}, \text{mmol} \cdot \text{g}^{-1}$</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Poly(AM-IA)/DAM-ZrW</td>
<td>8.0 ± 0.2</td>
<td>5.3</td>
<td>This work</td>
</tr>
<tr>
<td>P(AA-MA)/Al$_2$O$_3$SiO$_3$</td>
<td>&lt;0.5</td>
<td>0.12</td>
<td>Attallah, Allan, and Mahmoud (2015)</td>
</tr>
<tr>
<td>P(AM-AA-AN)/SiO$_2$-ZrO$_2$</td>
<td>8.0</td>
<td>0.24</td>
<td>Allan, Holiel, and Sanad (2014)</td>
</tr>
<tr>
<td>P(AM-IA)/ZrO$_2$</td>
<td>8.0</td>
<td>0.32–1.62</td>
<td>Hamoud et al. (2014)</td>
</tr>
<tr>
<td>P(AA-AN)/Ti-vanadate</td>
<td>6</td>
<td>1.55</td>
<td>El-Gamal et al. (2009)</td>
</tr>
<tr>
<td>Raw olive pomace</td>
<td>5.4</td>
<td>360</td>
<td>Omar and Attia (2013)</td>
</tr>
<tr>
<td>Carbonized olive pomace</td>
<td>3.6</td>
<td>565</td>
<td>Omar and Attia (2013)</td>
</tr>
<tr>
<td>PATiW composite</td>
<td>–</td>
<td>1.632</td>
<td>El-Naggar et al. (2012)</td>
</tr>
<tr>
<td>Sericite</td>
<td>7</td>
<td>0.05</td>
<td>Kim, Lee, and Jeon (2014)</td>
</tr>
<tr>
<td>Ion-imprinted polymer</td>
<td>6</td>
<td>0.243</td>
<td>Zhang, Xu, and Yan (2010)</td>
</tr>
<tr>
<td>Ceiling tiles</td>
<td>4.95</td>
<td>0.004</td>
<td>Miah et al. (2010)</td>
</tr>
<tr>
<td>Na-alginate</td>
<td>6</td>
<td>0.607</td>
<td>Khotimchenko et al. (2014)</td>
</tr>
<tr>
<td>Ca-alginate</td>
<td>6</td>
<td>0.479</td>
<td>Khotimchenko et al. (2014)</td>
</tr>
</tbody>
</table>

Figure 13. Adsorption performance of $^{134}$Cs$^+$ in solutions of some mineral acidic by synthesized poly(AM-IA)/DAM/ZrW composite.

Figure 14. Adsorption performance of $^{134}$Cs$^+$ in solutions of some organic acids by synthesized poly(AM-IA)/DAM/ZrW composite.

Figure 15. Adsorption performance of $^{134}$Cs$^+$ in solutions of some salts by synthesized poly(AM-IA)/DAM/ZrW composite.

Figure 16. Adsorption performance of $^{134}$Cs$^+$ in varied acetone solutions by synthesized poly(AM-IA)/DAM/ZrW composite.

### Conclusion

In this work, poly(AM-IA)/DAM/ZrW composite material was synthesized and characterized by different measurements (FTIR, XRD, TGA, DTA, SEM, and BET). It summarizes that: (i) the optimum conditions for preparing the composite material are 0.01 g DAM as a crosslinker, a co-monomer concentration 20%, a co-monomer composition (AM-IA) (12:88), and 0.03 g ZrW with gamma irradiation dose 30 kGy; (ii) the maximum adsorption performance of the composite is 93% for radiotracer and ions of $^{134}$Cs$^+$ in aqueous solution at pH 8 after 90 min; (iii) sorption process of Cs$^+$ ions was favored by the pseudo-second-order kinetic model and controlled by the Freundlich equation model, confirming the heterogeneous chemisorption process with adsorption capacity ($q_{\text{max}}$) of 5.29 mmol·g$^{-1}$; and (iv) applicability of the adsorbent was also examined to remove Cs$^+$ ions or its radioactive isotopes $^{134}$Cs$^+$ and/or $^{137}$Cs$^+$ from different solutions. It is interesting to
conclude that the prepared poly(AM-IA)/DAM/ZrW composite material could be used as a suitable adsorbent to immobilize the Cs\(^{+}\) ions or its radioactive isotopes from an alkaline waste solution associated with nuclear medical, industrial, or research centers.

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References


