acrylic acid)-silicon titanate has a semicrystaline nature. This crystalinity may by assigned to the presence of inorganic species in P(AM-AA) [127].

3.2.3. X-Ray fluorescence spectroscopy (XRF)

X-ray fluorescence is a tool used in this work for the determination of the inorganic ion exchanger that impregnated with the solid resin. The rasin was grind in a mill to very fine polymer, then mixed with a pindyer (Polyvinyl methaylate) to facilate the pressing process. The pressed sample in sample holder was exposed to X-ray and the X-ray spectrometer data determined are represented in Table (4). Table (4) shows that the present of silicon and titanium impregnated in the resin are 4.19 and 30.07%, respectively.

3.2.4. Chemical stability

Chemical stability of poly(acrylamide–acrylic acid) and poly(acrylamide-acrylic acid)—silicon titanate resins were tested in water and different concentrations (0.1, 1, 2, 3 and 5 M) of nitric and hydrochloric acids media. The results are summarized in the Table (5). The data in the Table (5) show that both resins are very stable in water and sparingly soluble in nitric and hydrochloric acids up to 5 M. Also, the data in Table (5) indicated that the chemical stability of the resin was increased by impregnation of silicon titanate in-situ P(AM–AA) and the stability of the resin in HCl is higher than in HNO₃ medium.

3.2.5. Effect of pH on the dissociation of the resin

The effect of pH of the medium on the dissociation of silicon titanate ion exchanger from the resin was studied by analyzed the solid of the resin before and after contact with the solution of different pH using X-ray fluorescence spectrometer. The data are represented in Table (6) indicated that the dissociation percent of silicon and titanium from P(AM-AA)-SiTi resin are very low and below detection limit up to 3-4 pH values. That means P(AM-AA)-SiTi resin is stable in the low pH value and the dissociation of silicon and titanium appears in pH above 4.

3.3. Effect of NaOH Treatment

The treatment of poly(acrylamide-acrylic acid)— silicon titanate by different concentrations of NaOH was done by soaking of 100 mg of P(AM-AA)—SiTi in 5 ml NaOH in a shaker thermostat for 2 hrs. Figure (5) shows that the recovery of Zn²⁺ ion on the untreated P(AM-AAA)—SiTi is negligible and as the concentration of NaOH used in the treatment process increases, the % recovery of Zn²⁺ ion increased. This behavior can be explained as the following manner; the treatment of P(AM-AA)—SiTi resin with NaOH leads to break of the cross-linking of the resin and the active sides of the chain are opened and covert the resin to the Na- form. As the concentration of NaOH increased the percent of the cross-linking decreased and the percent recovery of Zn²⁺ ion increased as shown in Fig. (5). At higher concentration values of NaOH

(> 0.3 M) the swelling degree is very high and there is no solution remained in the bottle. According to the result obtained, P(AM-AA)-SiTi was firstly treated by 0.3 M NaOH before used in all further investigation.

3.4. Batch Investigations

3.4.1. Effect of contact time on sorption behavior of P(AM-AA)-SiTi

The effect of contact time on the removal percent (%) uptake of Cu²⁺, Cd²⁺, Ni²⁺ and Zn²⁺ ions on P(AM-AA)-SiTi was studied at 25±1°C in a shaker thermostat with batch factor (V/m) = 100 ml.g⁻¹. The data obtained are represented in Fig. (6) and indicated that the maximum removal percent for all mentioned cations was attained within 3 hrs. In all experimental studies, the separation process was carried out within 4 hrs.

3.4.2. Effect of batch factor (V/m)

Effect of the batch factor {the ratio of volume of solution (V) to the amount of resin (m)} on the efficiency removal of Zn²⁺, Cd²⁺, Cu²⁺ and Ni²⁺ ions by P(AM-AA) and P(AM-AA)-SiTi resins was optimized by shaking 10 ml (50 ppm) of Zn²⁺, Cd²⁺, Cu²⁺ or Ni²⁺ ion solutions with the amount of the resins varied from 20 to 500 mg in a shaker thermostat at 25±1°C. The results of the effect of batch factor are represented in Figs. (7 and 8). Figures (7 and 8) show that as the batch factor increased the percent recovery of Zn²⁺, Cd²⁺, Cu²⁺ and Ni²⁺ ions are decreased. Also Figs. (7 and 8) depict that 50 mg of P(AM-AA) and 100 mg of

P(AM-AA)-SiTi are sufficient for the quantitative removal of Zn²⁺, Cd²⁺, Cu²⁺ and Ni²⁺ ions from their aqueous waste solutions. These amounts of P(AM-AA) and P(AM-AA)-SiTi were used for further investigations. Figure (6) shows also that at V/m ratio less than 200 the swelling degree of P(AM-AA) is very high and there is no residue solution sufficient for the determination of the metal ion concentration.

3.4.3. Effect of pH of the medium

The effect of pH of the medium on the absorption of Zn²⁺, Cd²⁺, Cu²⁺ and Ni²⁺ ions on P(AM-AA)-SiTi was studied in the pH range of 1.0 to 8.0. Where at pH > 8.0, the metal ions tend to precipitate. Figs. (9 and 10) show the effect of pH on the % uptake and the loading of Cu²⁺, Zn⁺², Cd⁺² and Ni²⁺ ions by P(AM-AA)-SiTi. Figs. (9 and 10) show that the % uptake and the loading (mg/g) are increase with increase in the pH range from 1.0 to 2.91, 4.64, 3.51 and 2.52 for all Ni²⁺, Cu²⁺, Cd²⁺ and Zn²⁺ ions, respectively. Poly(acrylamide-acrylic acid)— silicon titanate contains acid groups through to be carboxylic and amide groups. These protons – donating groups are responsible for the cation exchange with a bond formation between the resin and metal ion [128-129]. The uptake of M²⁺ ion could be explanation in the following equilibrium:-

$$n^* COO^-, Na^+ + M^{2+} \iff *(-COO)_n M^{(2-n)^+} + n Na^+$$

 $n^* NH^-, Na^+ + M^{2+} \iff *(-NH)_n M^{(2-n)^+} + 2 Na^+$

Where n = 1 or 2 and water molecules are omitted.