Polymeric Composite Materials Based On Silicate; I-Synthesis, Characterization And Formation Mechanism

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Abstract
Magneso-silicate has been synthesized by precipitation technique. Polyacrylamide acrylic acid and polyacrylamide acrylonitrile impregnated with inorganic ion exchanger, magneso-silicate, have been synthesized by subjected co-monomers to gamma radiation initiated polymerization at radiation doses 25, 65 and 90 KGY. The structure features of composites were investigated by sequential x-ray fluorescence spectrometer, x-ray diffraction, differential thermal- thermogravemetric analyses and infrared spectroscopy. Formation mechanism for these composites were conducted and the results obtained showed that the polymerization process were carried out in the hydrocarbon chain by addition polymerization whereas impregnation of magneso-silicate into the polymeric composites were carried out by condensation polymerization.

Keywords: Magneso-Silicate, Co-Monomers, Gamma Radiation, Condensation Polymerization.

1- INTRODUCTION
Now inorganic ion exchange materials play an important role in analytical chemistry, based originally on their resistance to chemical attack as well as their thermal and radiation resistance [1,2]. Polymers can be synthesized through various techniques such as radical, cationic and anionic polymerization[3]. The structural, mechanical and thermal properties can be investigated through different kinds of characterization methods to determination of structure property relationships[3,4]. Recently, polymers have been applied in various fields such as automotive, construction, electronic, cosmetic and pharmaceutical industries due to its advantageous material properties. Functional polymers of photochromic[5], electrochromic[6] and optoelectronic[7] functions were developed recently. The use of polymers with tunable refractive properties as optical modulators, optical filters, or electro optic waveguide devices has been reported[8]. The development of new inorganic ion exchangers with characteristic properties is still need attention and their utility in diverse fields is yet to be explored. Synthetic ion exchangers are used on a wide range for different applications, ranging from environmental remediation [1,9,10], water softening [10], hydrometallurgy [10] and selective adsorption [11,12] to medical applications [13-15]. Different materials based on silicate salts and poly acrylamide acrylic acid silicon titanate were synthesized earlier by Abou-Mesalam et al. [1,11,16] and used for removal of some heavy metals from industrial and hazardous waste solutions.

In this work magneso-silicate(MgSi) as inorganic ion exchange material was synthesized using precipitation technique. Polyacrylamide acrylic
acidP(AM-AA), polyacrylamide acrylic acid magneso-silicate \{P(AM-AA)-MgSi\} and polyacrylamide acrylonitrile magneso-silicate \{P(AM-AN)-MgSi\} composites have been synthesized by gamma radiation initiated polymerization at radiation doses 25, 65 and 90KGY. The prepared composites were analyzed by different analytical techniques and a new ion exchange character was represented compared to the original ones.

2- EXPERIMENTAL

All chemicals and reagents used were of analytical grade.

2.1- SYNTHESIS OF MAGNESO-SILICATE COMPOSITE

Magneso-silicate ion exchange material was synthesized as reported earlier by Abou-Mesalam et al. [1,11,14] by the addition of equimolar solutions (0.5M) of magnesium chloride to sodium metasilicate dropwisely with volumetric ratio for (Mg/Si) equal 1.5 with continuous stirring in a water bath adjusted at 60±1°C. The mixed solutions were immediately hydrolyzed in demineralized water. Diluted ammonia solution was added to the mixture until complete precipitation attained. The precipitate formed was kept in the mother solution overnight, then washed several times with distilled water, and then washed by 0.1M HNO₃ to remove impurities and Cl⁻ ions. The precipitate was washed by distilled water to remove NO₃⁻ ions. After drying at 60±1°C, solid was poured in near boiling distilled water heated at 70±1°C to break the solid and remove air trapped inside the solid, then redried at 60±1°C. The obtained solid was ground and stored at room temperature.

2.2- SYNTHESIS OF MONOMER SOLUTIONS

The investigated monomer solutions, acrylamide (AM), acrylic acid (AA) and acrylonitrile (AN) were prepared by dissolving 10% of each monomer in deoxygenated water.

2.3- SYNTHESIS OF CO-MONOMER SOLUTIONS

The acrylamide (AM) monomer solution was mixed with an aqueous solutions of acrylic acid (AA) and acrylonitrile (AN) by dropwisely at constant stirring and room temperature with volumetric ratio equal unity for the preparation of (AM+AA) and (AM+AN) co-monomers, respectively. Then the (AM+AA) and (AM+AN) co-monomers were mixed with equimolar solutions (0.5M) of sodium metasilicate and magnesium chloride hexahydrate by dropwisely at constant stirring and room temperature with volumetric ratio (AM-AA-Mg-Si) and (AM-AN-Mg-Si) equal 1:1:1.5:1, respectively.

2.4- SYNTHESIS OF P(AM-AA), P(AM-AN), \{P(AM-AA)-MgSi\} AND \{P(AM-AN)-MgSi\} COMPOSITES

P(AM-AA), P(AM-AN), \{P(AM-AA)-MgSi\} and \{P(AM-AN)-MgSi\} composites were prepared by subjected mixtures of (AM-AA), (AM+AN), (AM+AA+Mg+Si) and (AM+AN+Mg+Si) co-monomers to gamma radiation at radiation doses 25, 65 and 90KGY with dose rate 1.05KGY/h. After irradiation, the obtained hydrogel was cut into small pieces with a stainless steel scissors, soaked in acetone for removal of unreacted monomers, washed with water [17], dried at 50°C, grained, sieved for different mesh sizes and stored at room temperature [18].

2.5- COMPOSITION AND CHARACTERIZATION OF SYNTHESIZED COMPOSITES

IR spectra of MgSi-P(AM-AA), P(AM-AN), \{P(AM-AA)-MgSi\} and \{P(AM-AN)-MgSi\} composites prepared at different radiation doses were carried out by mixing of the solid with KOH in ratio 1:5 and ground to a very fine powder. A transparent disc was formed in a moisture free atmosphere. The IR spectra were recorded using BOMEM FTIR spectrometer in the range 400–4000 cm⁻¹. The stoichiometry of the constituents in MgSi and polymeric composites based
tosilicate prepared at different radiation doses were determined using Philips sequential x-ray spectrometer-2400. The solid samples were ground to very fine powders and then mixed with H$_3$BO$_3$ as a binder to facilitate the pressing process. The mixture was pressed in a sample holder of 40mm diameter aluminum cups and pressed on pressing machine at 20 psi to produce a sample with the diameter of 40mm and 5 mm thickness. The concentrations of magnesium and silicone were measured according to Super-Q quantitative application program.

X-ray diffraction patterns of prepared composites were carried out using a Shimadzu XD-D1, X-ray diffractometer with Cu-K$_\alpha$ radiation tube source ($\lambda$=1.5406Å) and graphite monochromator operating at 30kV and 30mA. The measurements were done in 2$\theta$ ranges from 4 to 90 with scan speed 2$^\circ$/min.

Prepared composites (20 mg) were analyzed for DTA and TGA with sample holder made of Pt in N$_2$ atmosphere using a Shimadzu DTG-60H. The heating rate was maintained at 10$^\circ$C/min with using alumina powder as reference material.

3- RESULTS AND DISCUSSION

The scope of this study is the attempt to synthesize a high chemical stable inorganic, organic and composite ion exchange materials with high selectivity for some heavy metals. MgSi, P(AM-AA), P(AM-AN), {P(AM-AA)-MgSi} and {P(AM-AN)-MgSi} composites prepared at different radiation doses have been synthesized with complete characterization for ion exchange materials.

Magneso-silicate (MgSi) was prepared earlier in our laboratories by Abou-Mesalam et al.[11]. The formation mechanism of prepared MgSi can be represented as shown inscheme (1). Magneso-silicate material was obtained by substitution of two Na'ions by one Mg$^{2+}$ion and elimination of two molecules of NaCl.
bond between C atoms of (AM) and (AN), the polymerization in the chain occurred by addition polymerization[21]. (AM+AN) free radical was obtained. (AM+AN) free radical react with (AM-AN) co-monomer to form chain propagation. Finally, the chains were coupled with another (AM-AN) free radicals to obtain P(AM-AN) copolymer.

Scheme (2): Formation mechanism of polyacrylamide acrylic acid copolymer.

Scheme (3): Formation mechanism of polyacrylamide acrylonitrile copolymer.

The formation mechanism of {P(AM-AA)-MgSi} composite can be represented as shown in scheme (4), the reaction started by converting of (AM) monomer to imine form in solution. Then imine form react with (AA+Mg+Si) to form (AM-AA-Mg-Si) co-monomer, and subjected to gamma radiation, breaking down was carried out for double bond of (AM) and (AA) and formation of covalent bond between C atoms of (AM) and (AA), the polymerization in the chain between (AM) and (AA) occurred by addition polymerization, where polymerization of (Mg-Si) in the chain was occurred by condensation polymerization [21] by elimination of OH⁻ of carboxylate group of (AA) with Na⁺ of Na₂SiO₃ to form ionic bond between O atom of carboxylate group and Si atom of Na₂SiO₃ and elimination of H⁺ of imine form with Cl⁻ of MgCl₂.6H₂O to form ionic bond between O atom of imine form and Mg atom of MgCl₂.6H₂O and elimination Na⁺ of Na₂SiO₃ with Cl of MgCl₂.6H₂O to form ionic bond between O atom of Na₂SiO₃ and Mg atom of MgCl₂.6H₂O to form (AM-AA-Mg-Si) free radicals. (AM-AA-Mg-Si) free radical react with (AM-AA-Mg-Si) co-monomer to form chain propagation. Finally, the chains were coupled with another (AM-AA-Mg-Si) free radical to obtain {P(AM-AA)-MgSi} composite.

The formation mechanism of {P(AM-AN)-MgSi} composite was shown in scheme (5), the reaction started by converting of (AM) monomer to imine form in solution. Then imine form react with (AN+Mg+Si) to form (AM-AN-Mg-Si) co-monomer, and subjected to gamma radiation, breaking down carried out for
double bond of (AM), and (AN), the polymerization in the chain between (AM) and (AN) occurred by addition polymerization, where polymerization of (Mg-Si) in the chain was occurred by condensation polymerization[29], by elimination of OH of imine form with Na+ of Na2SiO3 with formation of ionic bond between C atom of imine form and Si atom of Na2SiO3 and elimination Na+ from Na2SiO3 with Cl of MgCl2.6H2O with formation of ionic bond between O atom Na2SiO3 and Mg atom of MgCl2.6H2O and elimination of H+ of solution with Cl of MgCl2.6H2O and lone pair of electron of N atom of (AN) were bonded by ionic bond with Mg atom of MgCl2.6H2O to form (AM-AN-Mg-Si) free radical. (AM-AN-Mg-Si) free radical react with (AM-AN-Mg-Si) co-monomer to form chain propagation. Finally, the chains were coupled with another (AM-AN-Mg-Si) free radical to obtain {P(AM-AN)-MgSi} composite.

Scheme (4): Formation mechanism of polyacrylamide acrylic acid magnso-silicate composite

Scheme (5): Formation mechanism of polyacrylamide acrylonitrile magnso-silicate composite.
The prepared samples of P(AM-AA), P(AM-AN), {P(AM-AA)-MgSi} and {P(AM-AN)-MgSi} composites are hard granulating in nature suitable for use in column operations with yellow color.

IR spectra of MgSi ion exchanger was shown in Figure 1. (a), from this Figure six characteristic bands were observed in the regions 3150-3670, ~1652, 1000-1100, 903, 640 and ~470 cm⁻¹. The absorption band at 3150-3670 cm⁻¹ may be attributed to the stretching mode of water and OH groups absorbed on the composite [1,22,23]. The strong band appear at ~1652 cm⁻¹ represents the bending mode of water molecules absorbed on MgSi composite [1]. The broad absorption band at 1000-1100 cm⁻¹ is due to the metal-oxygen (Mg-O) bond [1]. The band at ≈ 903 cm⁻¹ may be due to the Mg-OH deformation vibration or overlapping of the Si-O and Si-OH, and Mg-O bonds in the structure [1,24]. The broad bands at 640 and 470 cm⁻¹ are assigned to Si-O-Mg and Si-O-Si bending vibrations, respectively [1,25].

IR spectra of P(AM-AA) copolymers prepared at radiation doses 25, 65 and 90 KGY was shown in Figure 2. (a), from this Figure two bands observed at 3450 and 3354 cm⁻¹ [26]. The first band may be attributed to the stretching mode of N-H bond of acrylamide and the second band attributed to the stretching mode of O-H of acrylic acid [27]. The band appeared at 3210 cm⁻¹ cross ponding to the stretching mode of H-O-H bonded of water molecules [28,29]. The bands appeared at (3090, 2950 and 2870 cm⁻¹) may be due to the stretching mode of C-H of acrylamide and acrylic acid [30]. The strong band appeared at 1720 cm⁻¹ attributed to the stretching mode of carbonyl group of acrylic acid [22,31]. Strong band appeared at 1550 cm⁻¹ attributed to the bending mode of N-H bond of acrylamide[32]. Two bands appeared at 1450 and 1370 cm⁻¹ may be attributed to the bending mode of C-H of acrylamide and acrylic acid [30,33]. Two broad bands appeared at 1213 and 1022 cm⁻¹ attributed to the bending vibration of C-N of acrylamide [33,34].

IR spectra of P(AM-AN) copolymers prepared at radiation doses 25, 65 and 90 KGY was shown in Figure 2. (b), from Figure represent two bands appeared at 3340-3450 and 3195 cm⁻¹ may be attributed to the stretching vibration of N-H bond of acrylamide [1,29]. Two bands observed at 2935 and 2875 cm⁻¹ can be attributed to the stretching mode of C-H of acrylamide and acrylonitrile [35,36]. The band appeared at 2790 cm⁻¹ may be due to the stretching mode of aldehyde group may be present by rearrangement in the structure [31]. The strong band appeared at 2244 cm⁻¹ due to the stretching mode of C≡N of acrylonitrile [35-38]. The band appeared at 1665 cm⁻¹ attributed to the stretching mode of carbonyl group of acrylamide [32]. Band appeared at 1605 cm⁻¹ may be attributed to the bending mode of N-H bond of acrylamide [32]. Two bands observed at 1450 and 1409 cm⁻¹ may be attributed to the bending mode of C-H of acrylamide and acrylonitrile [34-37]. Three bands appeared at 1316, 1182 and 1120 cm⁻¹ attributed to the bending vibration of C-N of acrylamide and acrylonitrile [32,34,37].

IR spectra of {P(AM-AA)-MgSi} composites prepared at radiation doses 25, 65 and 90 KGY was represented in Figure 2. (c), this Figure show that; broad band observed at 3460-3200 cm⁻¹ can be attributed to the stretching mode of water and OH group absorbed on the composites [1]. The two bands observed at 3460 and 3200 cm⁻¹ can be attributed to the stretching mode of N-H bond of acrylamide and H-O-H bonded of water molecules or O-H of acrylic acid respectively [27]. Two bands appeared at 2960 and 2875 cm⁻¹ may be due to the stretching mode of C-H of acrylamide and acrylic acid [29-31]. Weak band appeared at 2245 cm⁻¹ due to the stretching mode of C≡C bond may be present by rearrangement in the structure [29,36]. Two bands appeared at 1725 and 1675 cm⁻¹, the
former band may be due to the stretching mode of carbonyl group of acrylic acid
\([26,32]\), and the later band due to bending vibration of carbonyl group of acrylamide
or due to presence of imine group or O-H bonded water molecules absorbed on the
composite \([32,39]\). Band appeared at 1605 cm\(^{-1}\) for \{P(AM-AA)-MgSi\} at
radiation dose 90 KGY attributed to the bending mode of O-H bonded water
molecules absorbed on the composite \([1]\). The band appeared at 1571 cm\(^{-1}\) due
to the bending mode of N-H bond of acrylamide \([32]\). Two bands appeared at
1451 and 1413 cm\(^{-1}\) may be attributed to the bending mode of C-H of acrylamide
and acrylic acid \([31,33,36]\). Two bands appeared at 1331 and 1218 cm\(^{-1}\) for
compositate radiation doses 25 and 65 KGY, where at 1331 and 1173 cm\(^{-1}\) for
compositate radiation dose 90 KGY may be attributed to the bending mode of C-N of
acrylamide \([32]\). Band appeared at 1104 cm\(^{-1}\), this band reflect that metal oxygen bond
Mg-O \([1,17,18]\). Three bands appeared at 793, 611 and 465 cm\(^{-1}\). The first band may
be attributed to the of Mg-OH deformation vibration or overlapping of the Si-O and
Si-OH and Mg-O bonds in the structure \([1,25]\). The second band may be attributed
to Si-O-Mg bending vibrations \([25]\). The third band may be due to Si-O-Si bending
divbrations \([1,25]\). The bands at 1104, 793, 611 and 465 cm\(^{-1}\) indicated that
impregnation of Mg and Si in the polymeric resin, these results were agree with
data obtained from XRF as will be see later.

IR spectra of \{P(AM-AN)-MgSi\} composites prepared at radiation doses 25, 65 and
90 KGY was shown in Figure 2. (d), from this Figure broad band observed at
3475-3300 cm\(^{-1}\) can be attributed to the stretching mode of water and OH group
absorbed on the composite or N-H bond of acrylamide \([1,27]\). Two bands observed at
3190 and 2940 cm\(^{-1}\) can be attributed to the stretching mode of N-H bond of
acrylamide and C-H of acrylamide and acrylonitrile respectively \([34,36]\). Strong
band appeared at 2244 cm\(^{-1}\) due to the stretching mode of C≡N bond of
acrylonitrile \([36]\). Two bands appeared at 1661 and 1605 cm\(^{-1}\), the first band may
be attributed to the bending mode C=O group of acrylamide or due to presence of
imine group \([32]\), and the second band may be due to bending vibration of N-H bond of
acrylamide or O-H bonded water molecules absorbed on the composite
\([1,36]\). Two bands appeared at 1455 and 1413 cm\(^{-1}\) attributed to C-H of acrylamide
and acrylonitrile \([30]\). Band appeared at 1571 cm\(^{-1}\) may be attributed to the bending
mode of N-H bond of acrylamide \([32]\). Two bands appeared at 1451 and 1413 cm\(^{-1}\)
may be attributed to the bending mode of C-H of acrylamide and acrylic acid
\([34-36]\). Two bands appeared at 1340 and 1220 cm\(^{-1}\) for compositate radiation dose
25 KGY, where at 1340 and 1188 cm\(^{-1}\) for compositate radiation dose 65 and 90 KGY
may be attributed to the bending mode of C-N of acrylamide \([32]\). The band appeared
at 1040 cm\(^{-1}\) attributed to metal oxygen bond Mg-O \([1,17,18]\). Three bands appeared at
905, 636 and 460 cm\(^{-1}\). The first band may be attributed to the of Mg-
OH deformation vibration or overlapping of the Si-O and Si-OH and Mg-O bonds in
the structure \([1,25]\). The second band may be attributed to Si-O-Mg bending
vibrations \([1,25]\). The third band may be due to Si-O-Si bending vibrations
\([1,25]\). The bands at 1040, 905, 636 and 460 cm\(^{-1}\) indicated that impregnation of Mg
and Si in the structure of polymeric resin, these results were agree with data obtained
from XRF as will be see later.

X-ray diffraction patterns of MgSi composite was represented in Figure 1. (b), from this Figure it is clear that MgSi has crystalline structure \([1,40]\). These results were agree with the data obtained from XRD of composites materials treated at different heating temperature \([1,11,14]\). The crystallinity of the prepared materials slightly improved with the increase of heating temperatures from 50°C to 850±1°C, and there is a sharp
improvement of crystallinity occurs at 850±1°C.

Figure 1. (a) IR spectrum of magneso-silicate; (b) XRD of magneso-silicate.

Figure 2. (a) IR spectrum of P(AM-AA); (b) P(AM-AN); (c) [P(AM-AA)-MgSi]; (d) [P(AM-AN)-MgSi] at different radiation doses.

Figure 3. (a), show that XRD of P(AM-AA) copolymers prepared at radiation doses 25, 65 and 90KGY. This figure indicated that P(AM-AA) copolymers have amorphous structure and these results were similar to the data obtained from XRD of polyacrylamide-co-acrylic acid prepared by Hassan, et al. [31]. In addition, the crystalline character of the prepared samples was increased with radiation doses from 25 to 90KGY. Figure 3. (b) shows XRD patterns for P(AM-AN) copolymers prepared at radiation doses 25, 65 and 90KGY. From this figure it is clear that the sample prepared at radiation dose 25 KGY has crystalline structure, and these results were similar to the data obtained from XRD of
potassium hexacyano cobalt (II) ferrate (II) polyacrylonitrile (KCFC–PAN)[41], where samples prepared at radiation doses 65 and 90 KGY have an amorphous structure. Figure 3, (c and d) show XRD patterns of {P(AM-AA)-MgSi} and {P(AM-AN)-MgSi} composites prepared at radiation doses 25, 65 and 90 KGY, respectively. From these figures it is clear that P(AM-AA)-MgSi and P(AM-AN)-MgSi have crystalline structure and these results were similar to the data obtained from XRD of polyacrylamide titanium tungstophosphate [19,42], and potassium hexacyano cobalt (II) ferrate (II) polyacrylonitrile (KCFC–PAN)[41]. In addition, the crystalline character of {P(AM-AA)-MgSi} samples were decreased with radiation dose from 25 to 90 KGY, where the samples prepared at radiation doses 25 and 65 KGY have crystalline nature, where sample prepared radiation dose 90 KGY have semi crystalline nature. On the other hand, the crystalline character {P(AM-AN)-MgSi} was decreased with radiation doses from 25 to 65 KGY then increase from 65 to 90 KGY, where the sample at radiation dose 65 and 90 KGY has amorphous and semi crystalline natures, respectively.

Differential thermal and thermogravimetric analyses (DTA&TGA) play a vital role in studying the structure and the properties of any materials where it has been widely used to investigate the decomposition characteristics of materials. DTA and TGA data were used here to provide an alternative model for the kinetics of the composite degradation. For all investigation studies of the composites the rate of heating is 10°C/min[17,34,42,43], and the data were tabulated in Tables 1 and 2. Table 1 was represented the data obtained for DTA and TGA analyses for inorganic ion exchange material MgSi and organic polymers P(AM-AA) and P(AM-AN) radiated at different radiation doses 25, 65 and 90 KGY. The data obtained are supporting the fact that MgSi have a good thermal stability comparing with the other inorganic ion exchangers and the weight
loss of MgSi when the sample calcinated at 800 °C equal to 33.4%[1].

DTA and TGA of P(AM-AA) and P(AM-AN) copolymers prepared at radiation doses 25, 65 and 90 KGy were represented in Table 1 and indicates the process was occurring via five stage process and the weight loss are continued up to 500 °C. The weight loss for P(AM-AA) and P(AM-AN) prepared at radiation doses 25, 65 and 90 KGy with the heating temperature indicates that a (67.15%, 100% and 68.3%) for P(AM-AA) and (56.98%, 61.3% and 25.5%) for P(AM-AN) when the samples are calcinated at 500 °C[19].

The elemental analyses of magneso-silicate and polymeric composites based on silicate prepared at different radiation doses were measured using XRF and tabulated in Table 3; the measured data is confirmed that impregnation of magneso-silicate in polymeric composites have good thermal stability comparing with the other inorganic ion exchangers. The weight losses of P(AM-AN)-MgSi prepared at radiation doses 25, 65 and 90 KGy with the heating temperature equal to 74.2%, 80.8% and 83.9% when the sample is calcinated at 800 °C[19].

4- CONCLUSION
Magneso-silicate (MgSi) has been synthesized by precipitation technique. P(AM-AA), P(AM-AN), {P(AM-AA)-MgSi} and {P(AM-AN)-MgSi} composites have been synthesized by subjected co-monomers to gamma radiation initiated polymerization at radiation doses 25, 65 and 90 KGy. From the data obtained from the analytical techniques such as IR, XRD, TGA, DTA and XRF it is indicating that the impregnation of MgSi in {P(AM-AA)-MgSi} and {P(AM-AN)-MgSi} shows an improvement in thermal stability and these composite materials have crystalline nature suitable for column chromatographic applications[46].
Table (1): DTA and TGA analyses for MgSi, P(AM-AA) and P(AM-AN) materials.

| Composite | Radiation Dose, KgY | Temp. °C | Temp. Range °C | Peak | Description                                                                 | Ref. | Water Loss | % Total water loss |
|-----------|-------------------|----------|----------------|------|------------------------------------------------------------------------------|------|------------|-------------------|
| MgSi      | -                 | 128.35   | 43-192         | Endo.| Crossbedding loss of free water                                              | 1,47 | 16.2      |                   |
|           | -                 | 257.2    | 192-283        | Exo. | Due to decomposition of residual OH-groups and condensation of non-bonded oxygen | 48   | 4.45       |                   |
|           | -                 | 501.16   | 283-800        | Endo.| The loss of chemical bond water                                              | 1,47 | 12.79     |                   |
| P(AM-AA)  | 25                | 69.29    | 44-125         | Endo.| Loss of external water molecule                                              | 49   | 5.95       |                   |
|           |                   | 219.88   | 125-240        | Exo. | Removal of the water of crystallization for P(AM-AA)                         | 50   | 8.94       |                   |
|           |                   | 338.27   | 240-384        | Endo.| Probably caused by the dehydration of carboxylic acid and decarboxylation or the loss of chemical bond water | 1,47 | 28.1       |                   |
|           |                   | 400.9    | 384-408        | Exo. | May be due to the complete decomposition of the organic part of the materials | 52-54 | 9.87      |                   |
|           |                   | 422.28   | 408-500        | Endo.| The chain scission in the main chain of poly acrylamide acrylic acid.        | 51   | 14.02      |                   |
|           | 65                | 86.6     | 40-137         | Endo.| Loss of external water molecule                                              | 49   | 8.6        |                   |
|           |                   | 219      | 137-253        | Endo.| Removal of the water of crystallization for P(AM-AA)                         | 50   | 22.06      |                   |
|           |                   | 362.47   | 352-378        | Exo. | May be due to the complete decomposition of the organic part of the materials | 52-54 | 25.64      |                   |
|           |                   | 392.56   | 378-500        | Endo.| The chain scission in the main chain of poly acrylamide acrylic acid.        | 51   | 51.76      |                   |
| P(AM-AN)  | 90                | 81.69    | 41-129         | Endo.| Loss of external water in the resin                                         | 49   | 4.08       |                   |
|           |                   | 222.45   | 129-248        | Endo.| Removal of the water of crystallization for P(AM-AA)                         | 50   | 9.79       |                   |
|           |                   | 274.7    | 248-311        | Exo. | Since the complete decomposition of the organic part of the materials       | 52-54 | 6.72       |                   |
|           |                   | 329.9    | 311-347        | Endo.| Due to dehydration of carboxylic acid and decarboxylation or the loss of chemical bond water | 1,47 | 28.3       |                   |
|           |                   | 350.08   | 347-367        | Exo. | The complete decomposition of the organic part of the materials             | 52-54 | 5.5        |                   |
|           |                   | 386.55   | 367-398        | Endo.| The loss of constitution water, which forms part of the crystalline network and it is generally presented like OH groups | 55-56 | 17.9       |                   |
|           |                   | 420.34   | 398-427        | Exo. | May be due to the complete decomposition of the organic part of the composites | 52-54 | 8.8        |                   |
|           |                   | 476.5    | 427-500        | Endo.| The chain scission in the main chain of poly acrylamide acrylic acid.        | 51   | 7.87       |                   |
| P(AM-AN)  | 25                | 80.5     | 54-133         | Endo.| Loss of external water present in the composite                              | 49   | 8.21       |                   |
|           |                   | 273.15   | 133-328        | Endo.| Due to removal of the water of crystallization for P(AM-AN) copolymer        | 50   | 16.46      |                   |
|           |                   | 372.48   | 328-384        | Exo. | Due to the complete decomposition of the organic part of the materials       | 52-54 | 12.5       |                   |
|           |                   | 309.57   | 384-418        | Endo.| The loss of constitution water, which forms part of the crystalline network and it is generally presented like OH groups | 55-56 | 12.3       |                   |
|           |                   | 360.44   | 418-452        | Exo. | The chain scission in the main chain of poly acrylamide acrylic acid         | 51   | 5.34       |                   |
|           |                   | 465.7    | 452-500        | Endo.|                                                                              | 2.05 | 2.05       |                   |
|           | 65                | 83.43    | 50-145         | Endo.| Loss of external water molecule                                              | 49   | 9.5        |                   |
|           |                   | 273.39   | 178-312        | Endo.| Removal of the water of crystallization for P(AM-AN)                         | 50   | 14.3       |                   |
|           |                   | 362.8    | 312-367        | Exo. | Due to the complete decomposition of the organic part of the polymer        | 52-54 | 8.27       |                   |
|           |                   | 378.22   | 367-400        | Endo.| The loss of constitution water, which forms part of the crystalline network and it is generally presented like OH groups | 55-56 | 14.28      |                   |
|           |                   | 469.76   | 400-500        | Endo.| The chain scission in the main chain of poly acrylamide acrylic acid.        | 51   | 14.68      |                   |
|           |                   | 85.63    | 57-137         | Endo.| Loss of external water molecule                                              | 49   | 4.78       |                   |
|           |                   | 240.47   | 137-286        | Endo.| Removal of the water of crystallization for P(AM-AN)                         | 50   | 6.04       |                   |
Table (2): DTA and TGA analyses for \{P(AM-AA)-MgSi\} and \{P(AM-AN)-MgSi\} composite materials.

| Composite Ion Exchanger | Radiation Dose, KGy | Temp. Range, °C | Peak Description | Ref. | Water Loss % | Total Water Loss |
|-------------------------|---------------------|-----------------|-----------------|------|--------------|-----------------|
| P(AM-AA)-MgSi           | 25                  | 80.92 - 38-139  | Endo. Caused by loss of external water molecule | 49   | 9.33         |                 |
|                         |                     | 263.72 139-313  | Endo. Removal of the crystalline water for \{P(AM-AA)-MgSi\} | 50   | 14.66        |                 |
|                         |                     | 396.14 340-343  | Exo. The complete decomposition of the organic part of the composite. | 52-54 | 1858         |                 |
|                         |                     | 434.18 434-517  | Endo. Probably caused by the dehydration of carboxylic acid and decarboxylation or the loss of chemical bond water | 1,4,7,49,51  | 17.71        |                 |
|                         |                     | 655.6 517-800   | Endo. The chain scission in the main chain of poly acrylamide acrylic acid. | 51   | 40.90        |                 |
|                         |                     | 92.17 67-164    | Endo. Loss of external water molecule | 49   | 6.48         |                 |
|                         |                     | 304.5 164-355   | Endo. Removal of the chemical bond water of \{P(AM-AA)-MgSi\} | 50   | 22.45        |                 |
|                         |                     | 375.15 355-409  | Exo. | | | |
|                         |                     | 429.19 409-455  | Exo. The complete decomposition of the organic part of the composite resin | 52-54 | 7.01 | 37.38 |
|                         |                     | 521.19 455-563  | Exo. | | | |
|                         | 65                  | 80.8 42-171     | Endo. Loss of free water molecule | 49   | 6.6 | | |
|                         |                     | 240.16 171-318  | Endo. Removal of the crystalline water for \{P(AM-AA)-MgSi\} | 50   | 14.32        |                 |
|                         |                     | 380.33 318-407  | Exo. The complete decomposition of the organic part of the composite ion exchangers | 52-54 | 19.85 | | |
|                         |                     | 420.55 407-431  | Endo. Due to loss of chemical bond water | 1.47 | 3.35 | | |
|                         |                     | 444.05 431-467  | Exo. Maybe due to the complete decomposition of the organic part of the materials | 52-54 | 5.88 | 38.64 |
|                         |                     | 503.78 467-800  | Exo. | | | |
|                         | 90                  | 76.26 57-132    | Endo. Loss of water molecule | 49   | 8.77 | | |
|                         |                     | 281.69 132-340  | Endo. Removal of the water of crystallization for \{P(AM-AN)-MgSi\} | 50   | 13.11        |                 |
|                         |                     | 372.13 340-398  | Exo. The complete decomposition of the organic part of the composites | 52-54 | 9.97 | | |
|                         |                     | 419.69 398-433  | Exo. The loss of interstitial water molecules | 1.47 | 4.78 | | |
|                         |                     | 515.58 434-577  | Exo. May be due to the complete decomposition of the organic part of the materials | 52-54 | 32.49 | | |
|                         |                     | 585.56 577-800  | Endo. The chain scission in the main chain of poly acrylamide acrylic acid. | 51   | 4.45 | | |
|                         |                     | 76.24 40-124    | Endo. Loss of free water molecule | 49   | 8.85 | | |
|                         |                     | 263.51 124-334  | Endo. Removal of the water bind molecules of \{P(AM-AN)-MgSi\} | 50   | 14.33 | | |
|                         |                     | 362.72 334-383  | Exo. The complete decomposition of the organic part of the ion exchange materials | 52-54 | 8.31 | | |
|                         |                     | 398.57 383-402  | Endo. The loss of chemical bond water | 1.47 | 2.86 | | |
|                         |                     | 448.38 402-472  | Exo. May be due to the complete decomposition of the organic part of the materials | 52-54 | 8.87 | 37.44 |
|                         |                     | 521.29 472-800  | Exo. | | | |
|                         |                     | 76.11 55-126    | Endo. Loss of non-bonded water molecule | 49   | 7.29 | | |
|                         |                     | 272.63 126-326  | Endo. Removal of the water of crystallization for \{P(AM-AN)-MgSi\} | 50   | 14.3 | | |
|                         |                     | 365.2 326-397   | Exo. May be due to the complete decomposition of the organic part of the materials | 52-54 | 12.73 | | |
| Temperature | Endo. Event | Exo. Event |
|-------------|-------------|-------------|
| 421.29      | 397-452     | The loss of chemical bond water |
| 527.2       | 452-800     | The complete decomposition of the organic part of the composite materials |
|             |             | 1.47 7.88 |
|             |             | 52.54 41.2 |
Table (3): Elemental analysis of magneso-silicate and polymeric composites based on silicate prepared at different radiation doses

| Composite               | Radiation Dose, KGY | Concentration, % |
|-------------------------|---------------------|------------------|
|                         |                     | Mg       | Si       |
| MgSi                    | ----                | 18.7     | 37.7     |
| P(AM-AA)-MgSi           | 25                  | 10.1     | 21.2     |
|                         | 65                  | 11.6     | 25.6     |
|                         | 90                  | 8.3      | 44.7     |
| P(AM-AN)-MgSi           | 25                  | 13.3     | 44.7     |
|                         | 65                  | 12.9     | 47.6     |
|                         | 90                  | 13.5     | 45.2     |

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