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Gamma Radiation-Induced Preparation of Polymeric Composite Sorbents and Their Structure Assignments

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This work deals with the preparation of some polymeric composites together with their structure assignment. The polymeric composites [P(AM-AA)—EDTANa₂, P(AM-AA)-mont and P(AM-AA)-KNiHCF] were prepared by gamma radiation-induced template polymerization of acrylic acid (AA) on polyacrylamide P(AM) as a template polymer in the presence of either ethylenediaminetetra acetic acid disodium salt (EDTANa₂), montmorillonite (mont) or potassium nickel hexacyanoferrate (KNiHCF) complex, respectively. The sorption capacity of these composites towards the Cu(II) ion were studied as a function of EDTANa₂, montmorillonite and KNiHCF weight ratio present in the final product. Also, their complexation behavior with Cu(II) was studied using FT-IR spectroscopy and the proposed structures of the formed complexes were assigned.

Keywords: preparation; polymeric composites; template polymerization; gamma radiation

1 Introduction

Composite materials are used today in scientific, technological and manufacturing fields. Composite material is a product comprising a combination of dissimilar constituents. Many investigators have prepared composite sorbents, consisting of inorganic sorbents and organic binding matrices to overcome the limitations of organic resins and inorganic adsorbents. It is known that polyacrylonitrile PAN and sulphonated polystyrene divinyl benzene are major organic binding matrices used in this trend (1, 2). Also different inorganic adsorbents such as; clay minerals, zeolites, metal hydrates, metal phosphates, metal oxides and etc. were handled in the preparation of composite sorbents.

Clay minerals were used with polymers to produce polymer clay composites. Traditionally, clay minerals were used as filling materials for the purpose of improving material properties and reducing product cost. Swelling or smectite clay minerals are very common naturally occurring minerals and reside in huge deposits around the world. Their crystal structure consists of two dimensional layers (thickness = 0.95 nm) formed by fusion of two silica tetrahedral sheets with an edge-shared octahedral sheet of either alumina or magnesia (3). Stacking of these layers leads to

Van der Waals gaps or galleries. These galleries (alternatively referred to as interlayer) are occupied by cations, typically Na⁺ and/or Ca⁺², which balance the charge deficiency that is generated by isomorphous substitution within the layers (e.g., tetrahedral Si⁺⁴ by Al⁺³ or octahedral Al⁺³ by Mg⁺²). Because these cations are not structural (called exchangeable cations), they can be easily replaced by other positively charged ions or molecules (4).

The most common smectite clay mineral is montmorillonite which has hydrophilic character, and so it is difficult to be dispersed homogeneously in organic polymers (5). Typical cation exchange capacity of montmorillonite clay is 70–110 meq/100 g (6). Polymerization of monomers could occur between layers of montmorillonite by overcoming the forces between these layers. Also small molecules may enter into clay galleries and become polymerized (7, 8).

There are many uses for the preparation of polymer clay composite as; monomer intercalation with in-sit polymerization and polymer intercalation with restacking of the exfoliated layers over the polymer (9). An inorganic-organic composite (superabsorbent polymer clay composite, SAPC) was prepared by intercalating polymer into expandable smectitic clay using gamma radiation-induced polymerization (10). Studies on the preparation of AM/bentonite composite have been performed using gamma radiation (11). Acrylic acid and sodium acrylate also used for preparation of SAPC by an electron-beam induced polymerization method (12).

Inorganic ion exchangers are characterized by their radiation chemical and thermal stability. Also, they have high

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sorption capacity and selectivity towards metal ions. Hence, inorganic sorbents could be applied successfully in the treatment of different waste streams (13, 14). On the other hand, these exchangers could not be obtained in granular form and have low mechanical strength. Consequently, they could not be used successfully in column operation (13).

Organic ion exchangers, especially copolymers could be prepared with definite grain size and resist abrasion and thus are useful in column operation. With regards to their stability, these exchangers are very sensitive towards high radiation doses and high temperature. To overcome such disadvantages, composites of organic and inorganic materials are prepared. These composites generally contain both an inorganic part (exchanger or sorbent) and organic binding matrix (generally polymers). In this instance, Cu, Co, Ni and Zn ferrocyanides were supported on silica (15), and the cellulose and wood powder were used to support Cu, Co, Ni, Fe and Zn hexacyanoferrates (16). The sorption capacity of the produced composites towards radiocesium was varied from 1.2 to 3.8 meqg⁻¹. Polyacrylonitrile PAN was used to support some transition metal hexacyanoferrate complexes and the produced composites were used for the separation of radiocesium from neutral and acidic aqueous solutions (17).

This work deals with the preparation of P(AM-AA)-EDTANa₂, P(AM-AA)-mont and P(AM-AA)-K₄Fe(CN)₆ composites by template polymerization of (AA) onto P(AM) as a template polymer in the presence of EDTANa₂, mont or K₄Fe(CN)₆ precipitate, respectively. Also, N,N'-methylene diacrylamide (DAM) is used as a crosslinker.

2 Experimental

2.1 Materials

N,N'-methylene diacrylamide (DAM), and acrylamide (AM), were obtained from Merck (Germany). Acrylic acid (AA) monomer was obtained from BDH (UK) and EDTANa₂ was an Oxford laboratory reagent. The clay mineral montmorillonite (mont) was supplied by AR Aldrich (Germany). Potassium ferrocyanide, K₄Fe(CN)₆ · 3H₂O and potassium ferricyanide, K₃Fe(CN)₆ were supplied by NICE (India).

2.2 Instruments

The FT-IR spectra were studied using a Bomem, Hartman and Borunz spectrometer model MB 157, Canada.

A Shimadzu UV/V-double beam spectrophotometer model UV/V-210A, Tokyo was used for measuring the concentration of Cu(II) ion in solutions before and after contact with the prepared polymeric composites.

3 Preparation of Polymeric Composite Sorbents

Firstly, polyacrylamide P(AM) was prepared by a gamma radiation-initiated polymerization of 10% acrylamide

monomer solution using a gamma radiation dose of 10 kGy at a dose-rate of 10 kGy/hr (18).

Secondly, the preparation of the polymeric composites was as follows: DAM aqueous solution (1%) was mixed with P(AM) gel (3%), then different concentrations of either mont, EDTANa₂ or K₄Fe(CN)₆ were added to the obtained mixture and vigorously stirred at room temperature for 2 h. Then, AA (10%) aqueous solution was added to the produced mixture, and then gamma irradiated with a radiation dose of 20 kGy. After irradiation, the solidified samples were cut into small pieces after precipitation in acetone, dried, and finally stored for further work.

The K₄Fe(CN)₆ complex was prepared as reported by Remes (19); a known concentration of nickel(II) chloride was added to an equal concentration of both potassium hexacyanoferrate(III), and potassium hexacyanoferrate(II) and mixed vigorously. The obtained (K₄Fe(CN)₆) precipitate was washed by distilled water and used in further work.

4 Sorption Capacity Determination

P(AM-AA)-EDTANa₂ and P(AM-AA)-mont were treated with 0.1 N NaOH to neutralize the carboxylic groups, while P(AM-AA)-K₄Fe(CN)₆ was used as prepared. The sorption capacity was determined using a batch technique; where 40 mg of the investigated composite sorbent was contacted with 20 ml of the metal-ion solution Cu(II) for 24 h. After phase separation, samples from the metal ion solution were analyzed for Cu(II) concentration spectrophotometrically. The sorption capacity was calculated as:

$$\text{Capacity}(q) = \frac{\text{Uptake \%}}{100} \times \frac{C_0 \times V}{m} \times Z(\text{meq/g})$$

$$\text{Uptake \%} = (1 - A/A_0) \times 100,$$

where A is the absorbance of the solution after contacting with the composite, A₀ is the initial absorbance of the solution, C₀(mol/l) is the initial concentration of Cu(II) in solution, V(ml) is solution volume, m (g) is the weight of composite used, and Z is the charge of the studied cation.

5 Results and Discussion

5.1 Studying the Capacity of P(AM-AA)-EDTANa₂ Composite

P(AM-AA)-EDTANa₂ was prepared by template polymerization of (AA) on P(AM) as template polymer in the presence of EDTANa₂ and DAM as a crosslinker. The sorption capacity of the produced composite P(AM-AA)-EDTANa₂ towards Cu(II) ion was studied as a function of the concentration of EDTANa₂ present in the final composite. The results given in Figure 1, show that the sorption capacity of P(AM-AA)-EDTANa₂ increases with increasing EDTANa₂ concentration up to 1 wt%. While a further increase in EDTANa₂ concentration leads to a decrease in

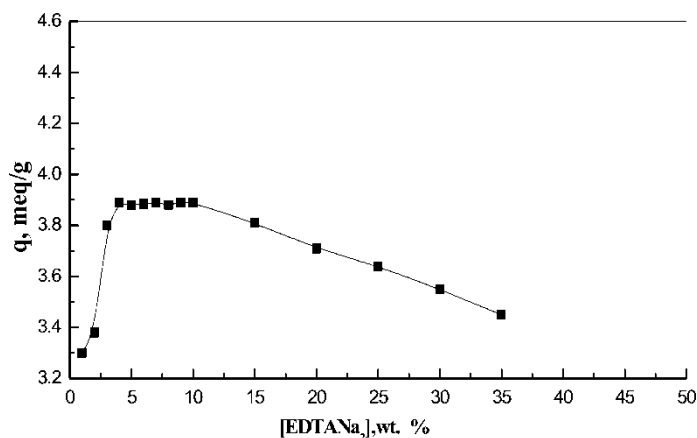


Fig. 1. Effect of EDTANa₂ ratio present in P(AM-AA)-EDTANa₂ on its sorption capacity towards Cu(II) ion.

the sorption capacity of the produced composite. As such, increasing EDTANa₂ concentration in the produced composite increases the number of free carboxylic and carboxylate groups available for the interaction with metal ions, while a further increase in EDTANa₂ concentration might increase the degree of crosslinking between the polymeric chains through interaction between EDTANa₂ and the functional groups of P(AM) and AA. Consequently, the number of the free carboxylic and amide groups of the produced composite, available for the interaction with the metal ions decreases. Hence, the sorption capacity decreases.

5.2 Studying the Capacity of P(AM-AA)-mont Composite

P(AM-AA)-mont was prepared by template polymerization of (AA) on P(AM) as a template polymer in the presence of montmorillonite and DAM as a crosslinker. The sorption capacity of the produced composite towards Cu(II) was studied as a function of montmorillonite concentration in P(AM-AA)-mont. The results given in Figure 2, show that the capacity of P(AM-AA)-mont increases with increasing the montmorillonite ratio up to 2 wt%, then decreases with a further increase of the montmorillonite concentration.

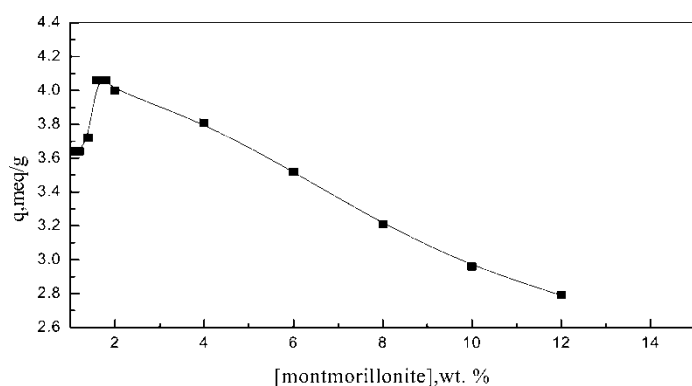


Fig. 2. Effect of montmorillonite ratio present in P(AM-AA)-mont on its sorption capacity towards Cu(II).

Increasing montmorillonite concentration leads to an increase in the exchangeable cations (Na⁺, Ca²⁺) of montmorillonite that can be easily replaced by other positively charged cations (4), while the observed decrease in the capacity of P(AM-AA)-mont with further increase in montmorillonite concentration may be attributed to the formation of a crosslinked structure in the polymer montmorillonite complex that inhibit the exchange of the metal cation.

5.3 Studying the Capacity of P(AM-AA)-KNiHCF Composite

P(AM-AA)-KNiHCF composite was prepared by the addition of KNiHCF precipitate during template polymerization of (AA) on P(AM) in the presence of DAM as a crosslinker. The sorption capacity of the produced composite towards Cu(II) ions was studied as a function of KNiHCF concentration in P(AM-AA)-KNiHCF. The results given in Figure 3 show that the capacity of P(AM-AA)-KNiHCF increases with increasing the KNiHCF concentration. This finding could be explained on the basis of increasing the exchangeable (K⁺ and Ni²⁺) ions present in the KNiHCF complex, which could be exchanged by other cations present in the solution (20).

5.4 Structure Assignment

The infrared spectral data of P(AM-AA)-EDTANa₂, P(AM-AA)-mont and P(AM-AA)-KNiHCF are given in Table 1. With respect to P(AM-AA)-EDTANa₂, the characteristic absorption bands for N-H stretching of amide groups, CH₂ stretching, COOH stretching, COO⁻ stretching and ester groups were obviously clear. This finding implies the presence of acrylamide, acrylic acid and DAM linked between polymeric chains.

It is also found that DAM acts as a crosslinker in the polymerization of acrylamide (AM), acrylamide-acrylic acid-dimethylaminoethylmethacrylate (AM-AA-DMAEM),

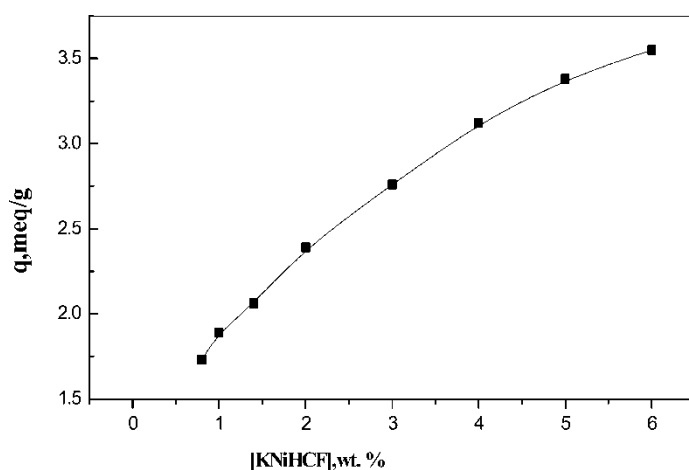


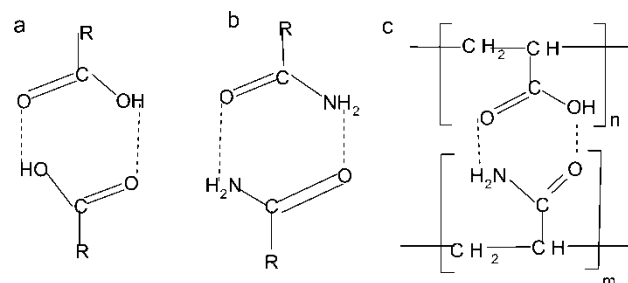
Fig. 3. Effect of KNiHCF ratio present in P(AM-AA)KNiHCF on its sorption capacity towards Cu(II).

Table 1. IR-spectral data for P(AM-AA)-EDTANa₂, P(AM-AA)-mont and P(AM-AA)-KNiHCF

Absorption bands of original groups, cm ⁻¹	P(AM-AA)-EDTANa ₂	P(AM-AA)-mont	P(AM-AA)-KNiHCF
Amide			
Free > NH stretching 3500, 3400	3438	3443	3438
Bonded > NH stretching 3350, 3180	3201, 3021		
> NH bend.(Amide II) 1650,1590			
>C=O stretching. (Amide I) 1650	1677		
Aliphatic			
-CH ₂ stretching 2926-2853	2923	2969	2918
-CH ₂ bend 1485-1445	1455	1460	1414
-CH ₃ bend 1470-1430, 1380			
Carboxylate			
-COO ⁻ 1610-1550 1400-1300	1543		
Ester			
>C=O stretching 1750-1735			1717
>C-O stretching 1300-1100	1171, 1017	1161	1182
Hydroxyl			
Free-OH stretching 3650-3590			
Bonded-OH stretching 3400-3200			
Carboxyl			
-COOH stretching 1725-1700	1734	1733	1717
-COOH bends 1420, 1300-1200	1156, 1270		
Free-OH stretching 3550			
Bonded-OH stretching 3300-2500	3201, 3021		
Amine			
C-N stretching 1410	1404		
-NH stretching 3000- 2700, 2700-2500			
Unsaturated Nitrogen Compound			
C≡N 2300-2000, 2260-2240			2089
Si-O		1049	
Si-O-Al		800	

acrylamide-acrylic acid (AM-AA), acrylamide-acrylic acid-acrylonitrile (AM-AA-AN), acrylic acid-acrylonitrile (AA-AN) (21–23).

There is also an absorption band characteristic for (C-N) of the amine groups. The appearance of the absorption band characteristic for the carboxylate groups indicates the

**Sch. 1.** Crosslinked structure formed through hydrogen bond between the function groups, a) carboxylate groups, b) amid groups, c) bonding between polymerized monomers and template.

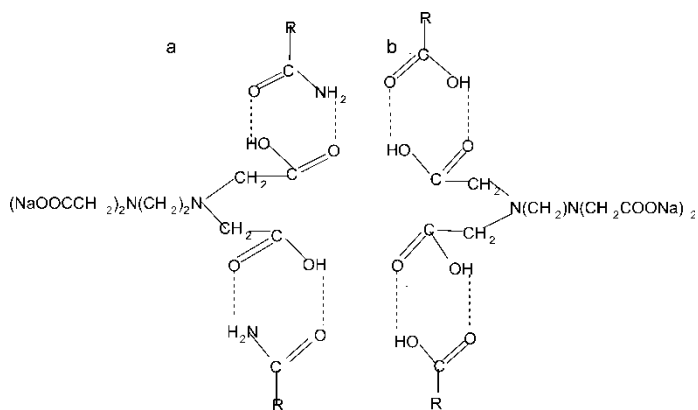
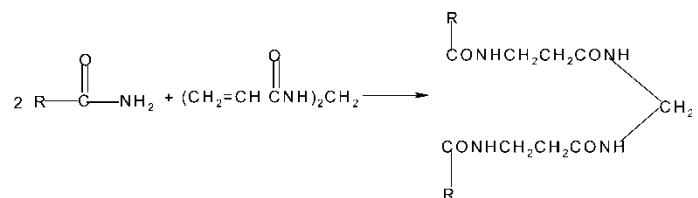
presence of linkage between EDTANa₂ and the polymeric chains through hydrogen bonding. The small shift produced in the peak positions could be due to the template polymerization.

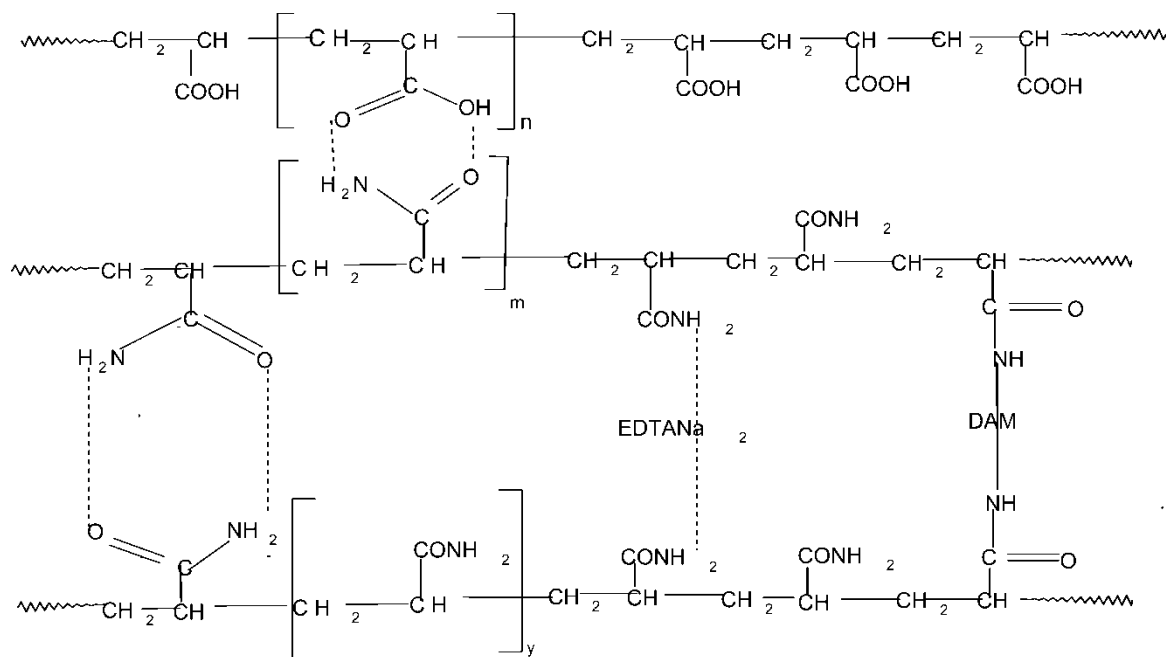
The suggested crosslinked structure of the polymeric composites could then be based on the following mechanisms:

Formation of a hydrogen bond between the functional groups of the two units of the polymeric chains, as shown in the Scheme 1.

Interaction of EDTANa₂ with the polymeric chains through hydrogen bond is shown in the Scheme 2.

Interaction of DAM with the functional groups of the polymeric chains (24) is shown in the Scheme 3.

**Sch. 2.** Crosslinked structure formed through interaction of EDTANa₂ with the polymeric chains; a) with amide groups, b) with carboxylic groups.**Sch. 3.** Crosslinked structure formed through interaction of DAM with amide groups.

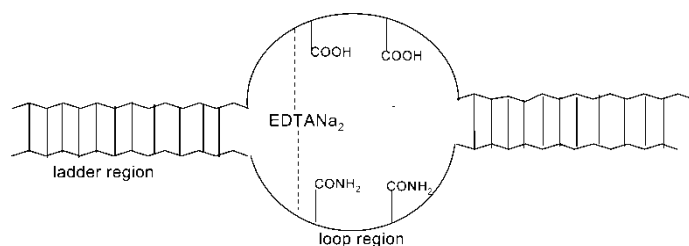


Sch. 4. Crosslinked structure formed through polymer complex interaction with template in presence of DAM and EDTANa₂.

Polymer complex formed through interaction between the daughters formed polymer and the template (24) as shown in Schematic Structure 4.

5.5 Formation of Ladder and Loop Regions

The ladder structures are formed as a result of the interaction between the monomer and the template through covalent bonds, while the loops are formed between the template and the monomer units in the regions in which the template and the monomer are separated. The bands at 1733 and 1677 cm⁻¹ could be attributed to the -COOH groups in the ladder and loop regions, respectively. This finding agrees with that of Baranovsky (25) for poly(methacrylic acid)/poly(ethylene glycol) in which the bands characteristic for the -COOH groups in the ladder and loop regions appeared at 1730 and 1710 cm⁻¹, respectively. The DAM and EDTANa₂ interact with the free functional groups in the loop regions as in Scheme 5.



Sch. 5. Formation of ladder and loop regions in polymer complex through interaction of free functional groups with EDTANa₂ and DAM.

Interactions between blocks (polymer complexes those formed between monomer and template) of daughter polymer (24–26) as in Scheme 6.

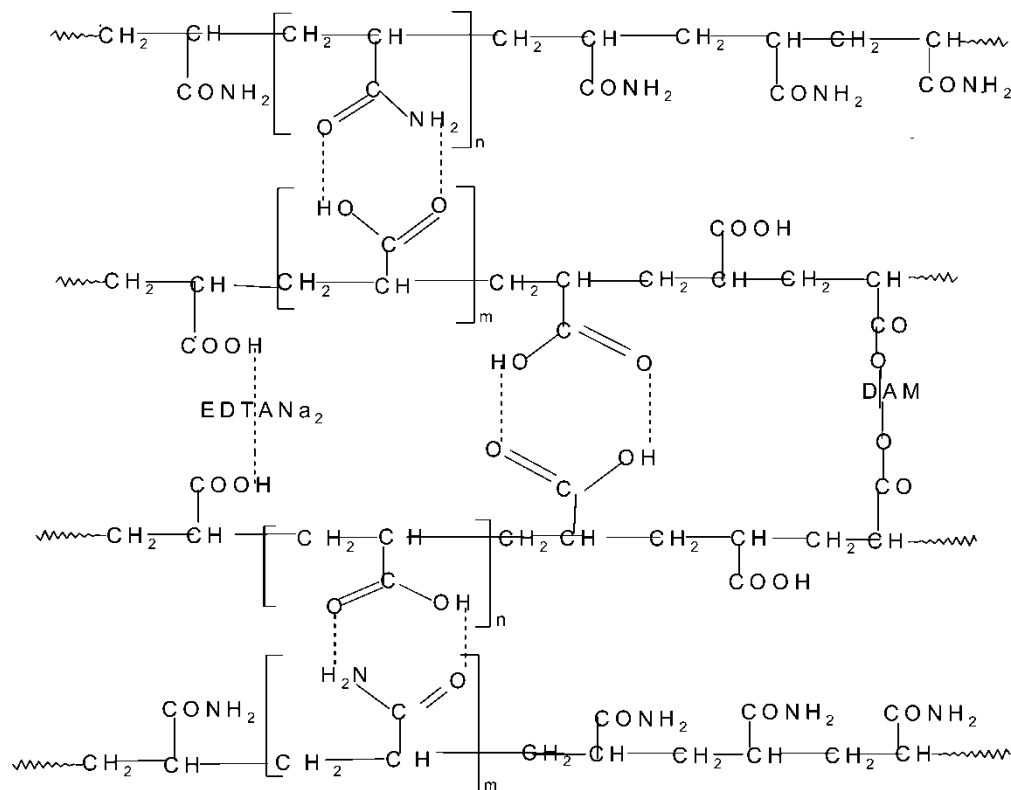
Formation of intermolecular crosslinked structures formed between polymeric chains as a result of imidation of amide groups by the effect of the gamma radiation on polyacrylamide in the aqueous solution (22) as in the Scheme 7.

Formation of polymeric materials through the interaction of free radicals formed by the effect of the gamma radiation on the template polymer P(AM) (27).

The montmorillonite clay contains a single octahedral sheet of [M(O;OH)₆, M: metal, e.g. Al, Mg, Fe] sandwiched between two tetrahedral sheets (SiO₄). The interlayer spaces between the sheets are occupied by exchangeable cations. The polymerization can take place within these interlayer spaces (28) as in Scheme 8.

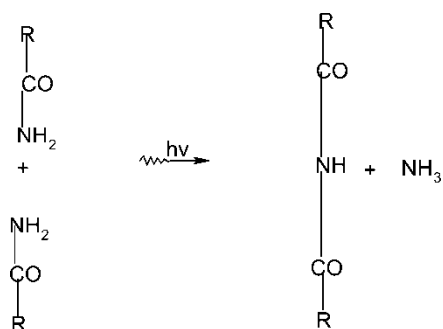
The IR- data of P(AM-AA)-mont show the bands characteristics for amide, carboxylic and ester groups. This implies the presence of acrylamide, acrylic acid units in the polymeric chains and also the DAM linked between polymer chains. There are also two bands characteristics for the Si-O-Al and Si-O groups at 800 and 1049 cm⁻¹, respectively. This finding indicates the intercalation of montmorillonite with the polymeric chains, which agrees with that of Gao (12) for intercalation of montmorillonite with polymeric chains of P(AM), where Si-O-Al and Si-O appeared at 799 and 1047 cm⁻¹, respectively.

The AM monomers undergo polymerization at 85°C, while if intercalated into montmorillonite, higher polymerization temperatures is required. This need for higher temperatures could be due to the restricted motion and intermolecular separation by the layered montmorillonite material which



Sch. 6. Crosslinked structure of polymer complex formed through the interaction of DAM and EDTANa₂ with blocks of polymer complex.

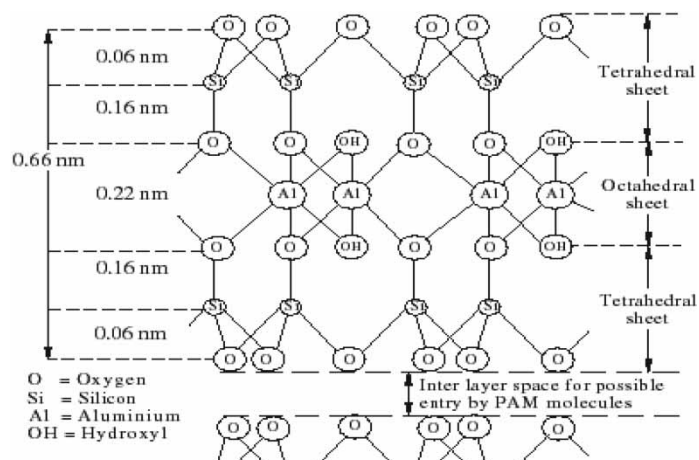
impedes cooperative interaction of monomeric species. Thus, either higher temperatures or other polymerization techniques must be applied such as irradiation with γ -rays (10), electron beams (29) or UV radiation (12). The kinetics and mechanisms of intercalation of acrylamide and montmorillonite are complex and are not known in detail. Ogawa et al. (30) assumed that during interaction of AM with montmorillonite, a coordination complex might be formed through bonding of interlayer exchangeable cations with surface oxygen atoms. Gao (12) suggests that AM could be intercalated into the interlayer spaces and on the surface of montmorillonite through a hydrogen bond.



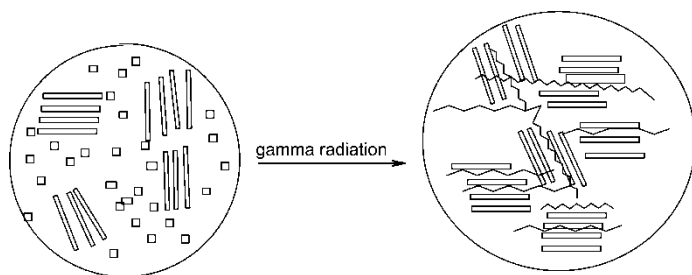
Sch. 7. Imidation of amide groups caused by gamma irradiation.



In this work, template polymerization of AA onto P(AM) in the presence of DAM as a crosslinker could take place in the interlayer space and on the surface of montmorillonite sheets after monomer and polymer intercalation into these interlayer spaces. The silicate layers were swollen by the molten P(AM), DAM and AA as follows:

Firstly, template P(AM) and monomer (AA) are intercalated into the interlayer spaces of montmorillonite. This intercalation could be due to the formation of the weak Van der



Sch. 8. Atomic arrangement in montmorillonite unit showing internal space for possible entry of P(AM) molecules.



Sch. 9. Formation of P(AM-AA)-mont,  montmorillonite sheets,  AA and P(AM).

Waals forces between the hydrated exchangeable cations and oxygen atoms of carbonyl groups. Also, a hydrogen bond may be formed between the amide ($-\text{CONH}_2$) and carboxylic ($-\text{COOH}$) groups of P(AM) and AA, respectively with the oxygen atoms in the silicate sheets of montmorillonite. Schemes (9,10-site A) clarify the intercalation of P(AM-AA) with montmorillonite.

Secondly, P(AM) and AA are bonded with the montmorillonite surface through a hydrogen bond with hydrated exchangeable surface cations. A hydrogen bond may also be formed between the carboxylic and the amide groups of AA and P(AM), respectively with the surface oxygen atoms of montmorillonite, as in Scheme 10-site B).

Finally, polymerization takes place by gamma radiation, and free polymer networks could be formed between the montmorillonite particles as in Scheme 10-site C). The produced polymeric composite P(AM-AA)-mont is highly crosslinked and also insoluble.

Crosslinked structures for P(AM-AA)-mont could be assigned, as mentioned before, in the case of P(AM-AA)-EDTANa₂.

The spectroscopic studies for P(AM-AA)-KNiHCF show the presence of the absorption bands characteristics for the amide, carboxylic and ester groups which implies the presence of acrylamide, acrylic acid unites in the polymeric

chains, as well as DAM linked between them. Also, there is an absorption band characteristic for ($\text{C}\equiv\text{N}$), indicating the presence of ferrocyanide complex linked with the polymeric chains. The absorption band characteristics for ($\text{C}\equiv\text{N}$) of KNiHCF complex appears at lower wave number (2089 cm^{-1}) which may be due to the intercalation of KNiHCF with the polymeric chains. The polymeric composite P(AM-AA)-KNiHCF is highly crosslinked and also insoluble. Also, crosslinked structures for P(AM-AA)-KNiHCF could be assigned as mentioned before in the case of P(AM-AA)-EDTANa₂.

6 Conclusions

Some polymeric composites (P(AM-AA)-EDTANa₂, P(AM-AA)-mont and P(AM-AA)-KNiHCF) could be prepared using gamma radiation induced template polymerization.

The polymeric composites have highly crosslinked structures, as a result of the presence of DAM and (EDTANa₂, mont or KNiHCF), as well as the gamma radiation effect.

The prepared composites have low solubility and no swelling due to the formation of crosslinked structures, as well as having high sorption capacity towards the metal ion (Cu(II)).

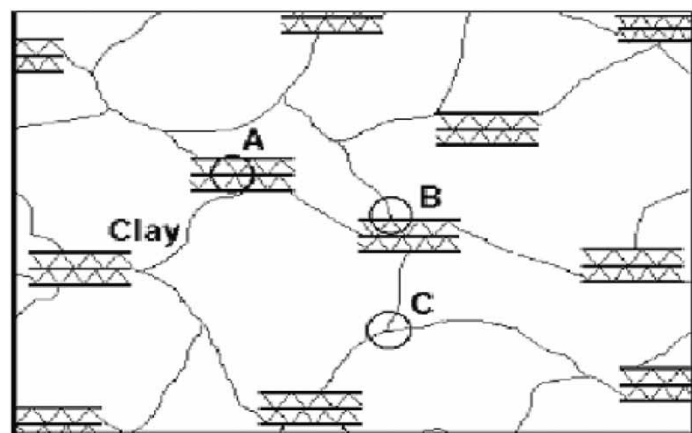
The assigned crosslinked structures of these polymeric composites reflect their higher stability towards temperature, radiation and chemicals.

The sorption capacity of these composites is highly affected by the weight ratio of EDTANa₂, mont and KNiHCF present in these composites.

The prepared polymeric composites could be used with high efficiency for metal ions separation, due to the presence of many functional groups as; amide, amine, carboxylic and carboxylate, as well as exchangeable cations.

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Sch. 10. Intercalation possibilities; A) polymer intercalated into the lamina of montmorillonite, B) polymer attached to the surface of montmorillonite and, C) free polymer network.

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