The Use of Wood Pulp and Radiation-Modified Starch in Wastewater Treatment

S. E. Abdel-Aal, Y. H. Gad, A. M. Dessouki

National Center for Radiation Research and Technology, Nasr City, Cairo, Egypt

ABSTRACT: Radiation-induced graft copolymerization of maize starch/acrylic acid has been performed. Also, natural byproduct wood pulp was used after chemical treatment for the removal of metal ions from the investigated wastewater. The surface and structure morphology of the wood pulp and starch/acrylic acid were investigated by scanning electron microscopy and infrared spectroscopy. The physical parameters, such as swelling, gel percentage, and grafting efficiency (%) of starch/acrylic acid copolymer, were studied. The factors affecting the abilities of the prepared materials for removing heavy metal ions and dyes from aqueous solutions were studied. It was found that the maximum metal uptake is in the following sequence: Fe^{3+} > Cr^{3+} > Pb^{2+} > Cd^{2+}. The adsorption capacity of such investigated metal ions increases with the increase of pH values. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 99: 2460–2469, 2006

Key words: radiation; grafting; wood pulp; wastewater treatment; metals; starch; acrylic acid

INTRODUCTION

Starch is an abundant, inexpensive, renewable, and fully biodegradable natural raw material. However, starch by itself is unsuitable for most uses as a plastic, because of its poor physical properties (mechanical properties, dimensional stability). Graft copolymerization is one of the efficient methods to modify natural[1–3] and synthetic polymers.[4–6] In this way, it is possible to modify many different properties in the starch, such as elasticity, sorbancy, ion-exchange capabilities, thermal resistance, and resistance to microbiological attack.[7–9] Starch has been used as a model substrate for graft investigations, mainly because of the ease with which vinyl monomers undergo grafting onto it. There are many reports upon chemical-[10–12] and radiation[13]-initiated grafting studies for the modification of starch. Starch graft copolymers are becoming increasingly important, because of their potential applications in industry. It has been used as hydrogels, flocculants, ion exchangers, superabsorbents, and so on.[14] Starch graft copolymers could be achieved primarily by free radical-initiated reaction.[15–18] The removal of toxic and polluting heavy metal ions from industrial effluents, water supplies, and mine waters has received much attention in recent years. It is well known [19] that heavy metal ions, such as Pb(II) and Cu(II) ions, released into the environment affect ecological life, owing to their tendency to accumulate in living organisms, and are highly toxic when absorbed into the body. Various methods, such as ion-exchange, reverse osmosis, as well as electrodialysis techniques, have been developed for the removal and recovery of heavy metal ions from sewage and industrial wastewater.[20] In spite of their removal effectiveness, they are often quite expensive. Traditional chemical precipitation can be envisaged, but the generation of precipitated bulky hydroxides and colloidal particles is a major disadvantage.

The search for an effective and economic method of removing toxic heavy metal ions requires the consideration of unconventional materials and processes. In this respect, many natural polysaccharides and their derivatives, containing various functional groups, may have some potential. Recently, chitin and its derivatives,[21–23] modified cellulose[24–26] as well as modified starch ethers,[27–29] have been studied with respect to their ability to remove heavy metal ions from aqueous solutions. Cellulosic materials are natural polymers and contain reactive hydroxyl groups in every constituent (hydroglucose unit). Therefore, cellulosic materials are very promising raw materials for the preparation of various functional polymers. The importance of chelating sorbents in chemical applications has been evident for a long period of time.[30]

The present work objective is to synthesize and characterize ion-exchange, starch-modified materials as a product of direct radiation grafting of acrylic acid (AAc) monomer onto starch, also, we used natural byproduct wood pulp after chemical treatment for the removal of pollutants, such as metals ions and dyes, from industrial wastewater.

Correspondence to: S. E. Abdel-Aal (yasser2uk@yahoo.com, yasser_hg@hotmail.com).
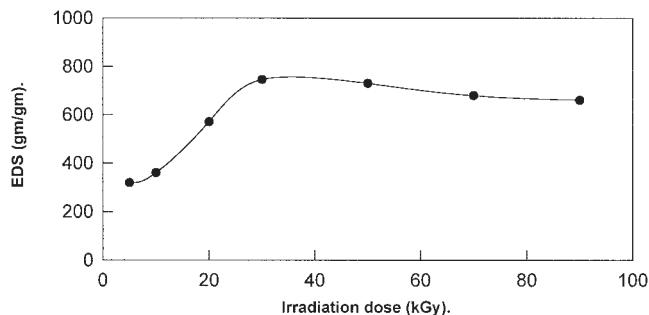
Figure 1 Effect of irradiation dose on the EDS (mg/g) of starch/AAc polymer, with starch : AAc (1 : 3) composition.

EXPERIMENTAL

Materials

The fibrous raw materials used in this work was de-pithed Egyptian wood pulp, obtained from El-Nasr Co. for sugar and pulp industry at Edfo, Egypt.

Reagent grade AAc of purity 99.9% (Merck, Germany), acrylamide of purity 98.9% (SD Fine, India). Other chemicals, such as solvents, inorganic salts, acids, *etc.*, were reagent grade and used as received.

Metal ions

$Cr(NO_3)_3 \cdot 9H_2O$ was supplied by laboratory BDH, Germany. $Pb(NO_3)_2$ and $Cd(NO_3)_2 \cdot 4H_2O$ was supplied by VEB laborchemie, Apolda, Germany and $Fe(NO_3)_3 \cdot 9H_2O$ was supplied by Merck.

Pretreatment of the fiber

The fibers were soaked in a detergent solution for 30 min, followed by extensive washing with tap water for 1 h, and finally with distilled water, and squeezed. The samples were then dried in a drying oven at 40°C for 4 h and then left to dry in air.

Alkaline treatment

The experiment was carried out in a round-bottom flask, fitted with a reflux condenser. The temperature was adjusted by means of a thermostat.

Alkaline pulping of wood pulp.

Portions of 80 g-depithed wood pulp were added to ethanol (50%), with liquor to wood pulp ratio of 10 : 1, and then, NaOH (2.5%) and anthraquinone (0.1%) were added to the mixture. The mixture was refluxed at a temperature of 85°C for 3 h. The pulp was extracted and soaked in 1 L cold ethanol (50%) overnight. After this, the pulp was washed with cold tap water to complete purity.

Bleaching

The experiment was carried out for wood pulp with 4% H_2O_2 in a single-stage bleaching process[31] at 105°C for 60 min.

Graft copolymerization by simultaneous irradiation

Ten grams of maize starch was mixed with 70 cm^3 of distilled water. The system was stirred and then heated at ~85°C, at the same time, for 1 h to form a paste-like slurry. The gelatinized starch was cooled at room temperature. A weighed quantity of monomers was added into the gelatinized starch, and the total volume was 100 cm^3. The mixture was stirred at 400 rpm at room temperature for 30 min. The gelatinized starch–monomer mixture was transferred into a 20-cm^3 glass and pulped with nitrogen gas for 10 min for each tube. It was closed tightly with foil and paraffin film, and then irradiated with γ rays. The reaction product was dried in a vacuum oven at 56°C for 24 h.

Effect of irradiation dose on starch modification

Irradiation of samples was carried out by using ^{60}Co γ ray at a dose rate of 4.25 kGy/h, and it is installed at the National Center for Radiation Research and Technology, Atomic Energy Authority, Egypt. Doses of 5, 10, 20, 30, 50, 70, 90 kGy were used to irradiate starch–AAc or starch–acrylonitrile mixture to observe the effect on gel percentage and water absorption.

Swelling study of the modified polymer

Starch-modified polymer samples were immersed directly in distilled water at room temperature to equilibrium (72 h); then, the swollen polymer was dried at 50°C to constant weight. The equilibrium degree of swelling (EDS) of the polymer was calculated as follows:

$$EDS = (W_e/W_d) \times 100 \qquad (1)$$

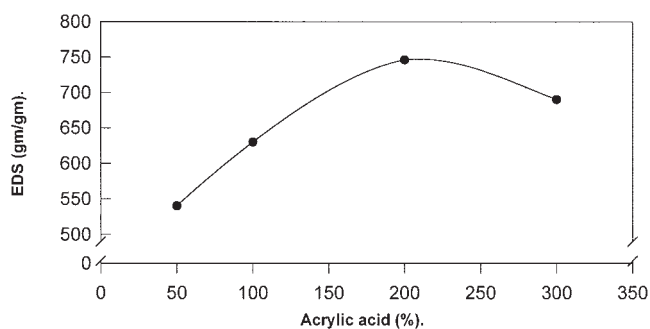**Figure 2** Effect of AAc (%) on the EDS (mg/g) of starch/AAc polymer at total irradiation dose of 30 kGy.
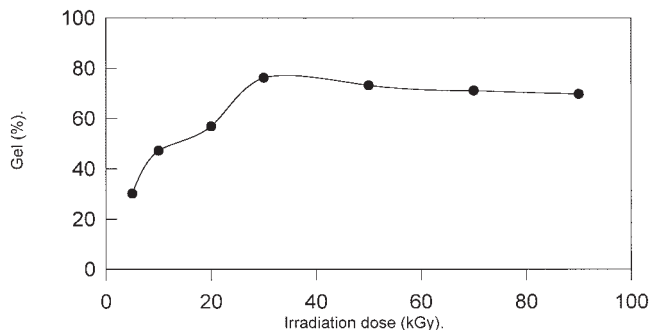
Figure 3 Effect of irradiation dose (kGy) on the gel (%) of starch/AAc polymer and starch : AAc (1 : 3) composition, at a dose rate of 4.25 kGy/h.

where W_e is the weight of polymer at equilibrium and W_d is the weight of the dried gel before swelling.

Gel fraction

The starch-modified polymer samples were put into a stainless net of 200 mesh, and then, the sol was extracted in distilled water at 120°C for 48 h, and then, washed with distilled water three times. The remaining gel was dried to constant weight at 50°C. Gel fraction was measured gravimetrically as follows:

$$\text{Gel fraction (\%)} = (W_g/W_0) \times 100 \qquad (2)$$

where W_g is the weight of the dry gel after extraction and W_0 is the initial weight of the dry polymer.

Grafting efficiency

Grafting efficiency % (GE) was calculated from the increase in weight of starch after grafting in the following manner:
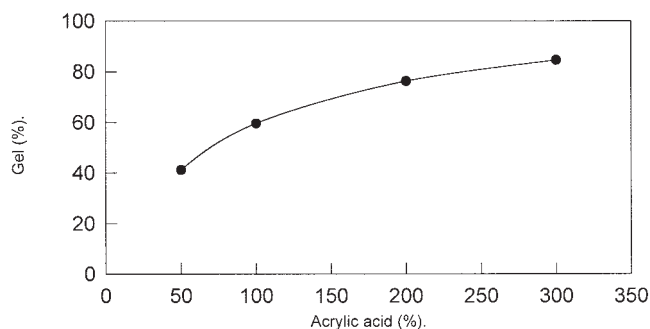

Figure 4 Effect of AAc (%) on the gel (%) of starch/AAc polymer, at a dose rate of 4.25 kGy/h and irradiation dose of 30 kGy.
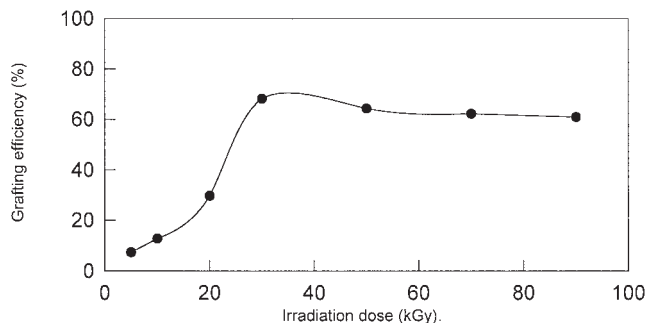

Figure 5 Effect of irradiation dose (kGy) on the GE of starch : AAc monomer, with starch : AAc (1 : 3) composition, at a dose rate of 4.25 kGy/h.

$$\text{GE (\%)} = \frac{\text{Weight of polymer grafted}}{\text{Weight of polymer grafted} + \text{Weight of homopolymer}} \times 100$$

$$(3)$$

Adsorption of metal ions

Heavy metal adsorption from the aqueous solution was studied in batch adsorption experiments. The effect of initial pH of the solution, metal ion concentration, and monomer percentage of starch-modified polymer on adsorption was studied. For batch tests, 0.5 mg of the starch-modified polymer or wood pulp was shaken with 50 mL of aqueous metal ion solutions, with a known concentration, at room temperature and at different pH values. Blank trials, without the addition of the starch-modified polymer or wood pulp, were carried out for each tested pollutant concentration. After the predetermined adsorption time, the polymeric or wood pulp adsorbents were separated from the adsorption media, and the analysis for heavy metal ions in the aqueous phase were performed using atomic absorption instrument (Unicam Model Solar 929). The adsorption capacity was calculated from the following expression:
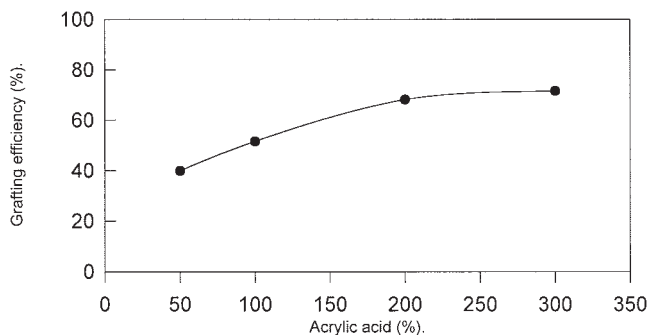

Figure 6 Effect of AAc (%) on the GE of starch/AAc polymer, at a dose rate of 4.25 kGy and irradiation dose of 30 kGy.
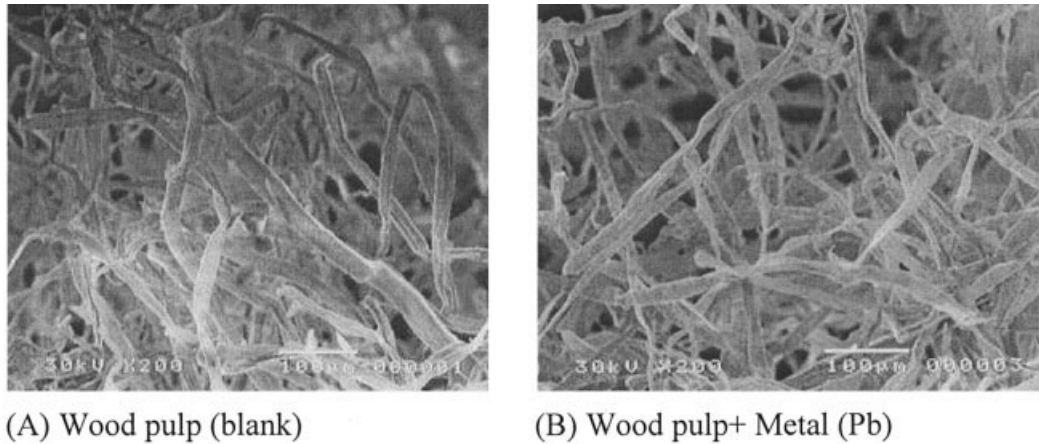
(A) Wood pulp (blank)

(B) Wood pulp+ Metal (Pb)

Figure 7 SEM of (A) wood pulp (blank) and (B) wood pulp + metal (Pb).

$$x/m = kC_e^{1/n} \qquad (4)$$

where x/m is the equilibrium sorbed concentration of the solute (mg/g) and C_e is the equilibrium aqueous concentration of the solute (mg/L). k and n are the fitted parameters determined from nonlinear regression.

Modified starch characterization

FTIR analysis

Infrared spectra (FTIR) of polymer samples with KBr pellets were obtained from FTIR spectrometer (Perkin–Elmer, 1725).
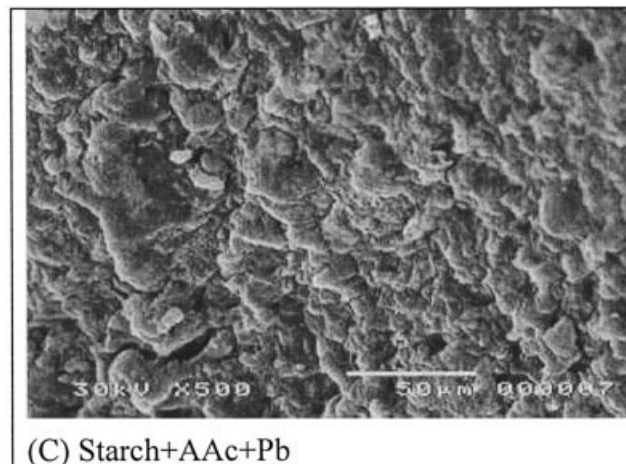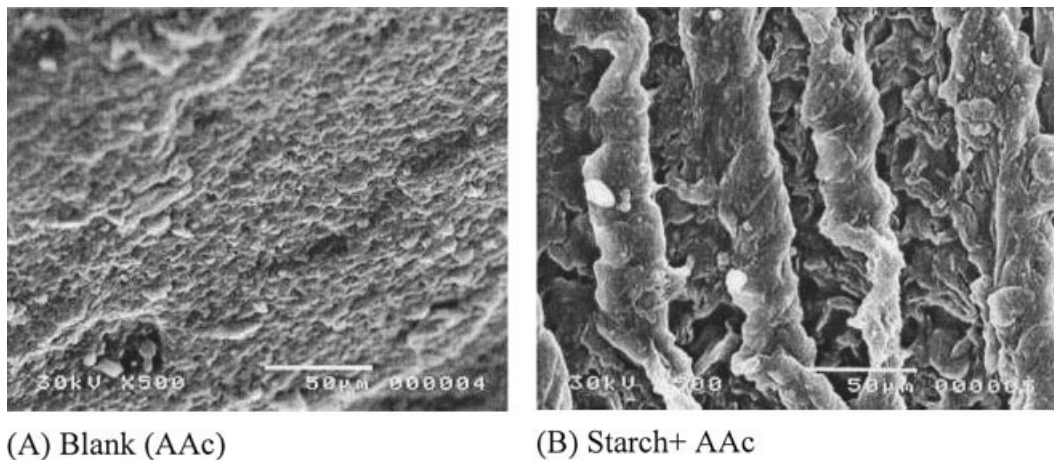

(A) Blank (AAc)

(B) Starch+ AAc

(C) Starch+AAc+Pb

Figure 8 SEM of (A) blank AAc, (B) starch + AAc, and (C) starch + AAc + Pb.
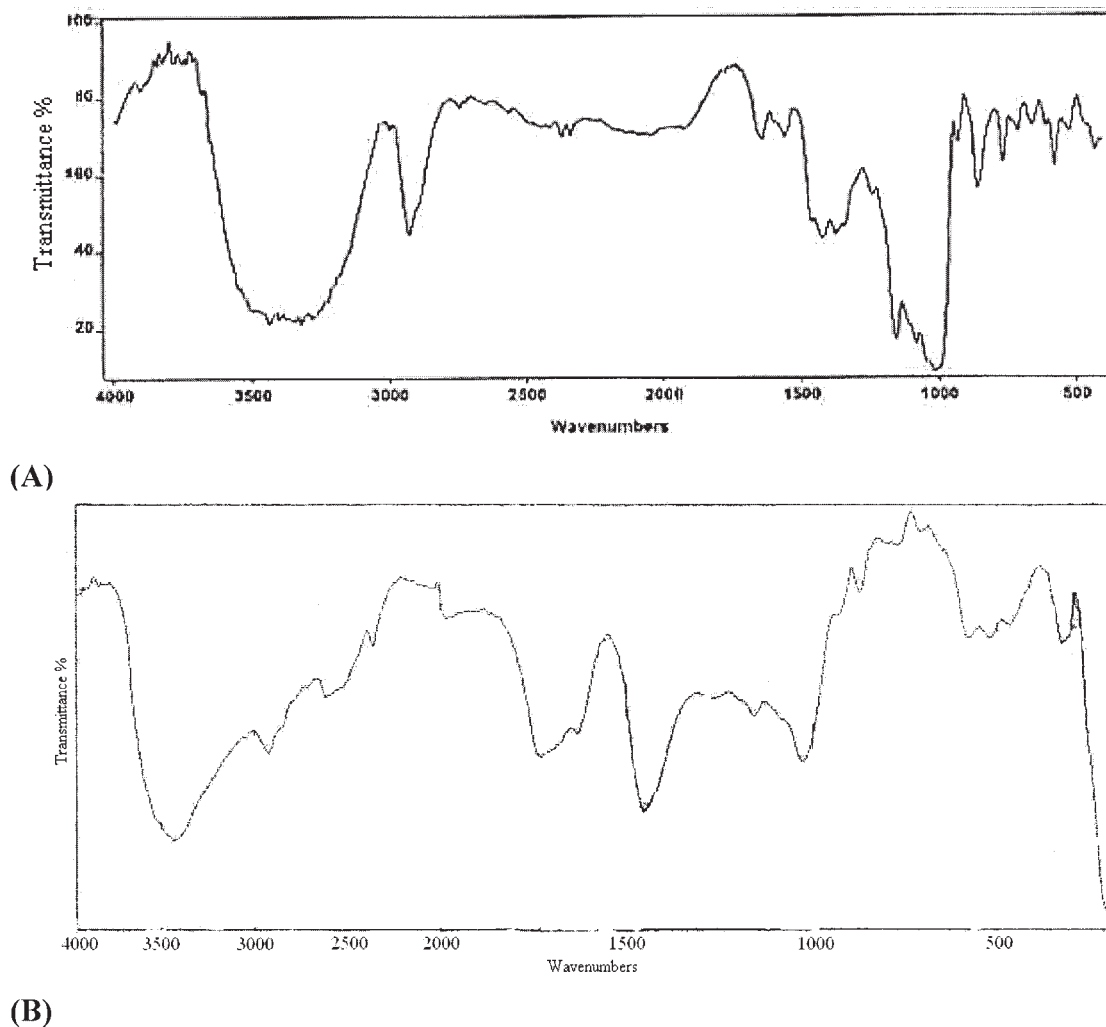
(A)

(B)

Figure 9 FTIR of (A) starch (blank) and (B) starch/AAc.

Scanning electron microscopy

The surface topography of the wood pulp and starch/ AAc materials was studied using JEOL Scanning Electron Microscope (SEM)-25 (Japan). Before the examination, the starch/acrylic and cellulosic materials were dried and coated with gold, under sputter.

RESULTS AND DISCUSSION

Swelling behavior

The change of swelling behavior of starch-*g*-AAc polymer, with the content of AAc in the polymeric system, and the radiation dose is shown in Figures 1 and 2. EDS of the prepared polymer decreased with the dose reasonably and increased with the addition of AAc, because of the high hydrophilicity of AAc and the poor hydrophilicity of starch.

Gel fraction percentage

The changes in the gel fraction of the prepared polymer with the dose as well as the content of AAc in starch-*g*-AAc

are shown in Figures 3 and 4. The gel fraction increased and then leveled off around 30 kGy, after which it decreased slightly because of the degradation of the polymers. The gel fraction increased with the increase in the content of AAc in the polymer formed. The results also showed that part of the starch could not be removed by extraction, *i.e.*, there were two kinds of starch in the modified polymer after irradiation. One was entrapped in the polymer, which could be removed easily by extraction, and the other could not be removed, as the latter grafted with starch in the prepared polymer. Polysaccharide and synthesized polymer hydrogels have been prepared and studied widely, and many researchers suggested that there was a chemical reaction between polysaccharide and synthesized polymer molecules, because saccharide groups contain two reactive groups at C2 and C6 positions, but up to now no related research was reported.[32–35] (Starch is a kind of polysaccharide.)

Grafting efficiency

The suitable monomer percentage varies from one system to another, depending on the starch substrate
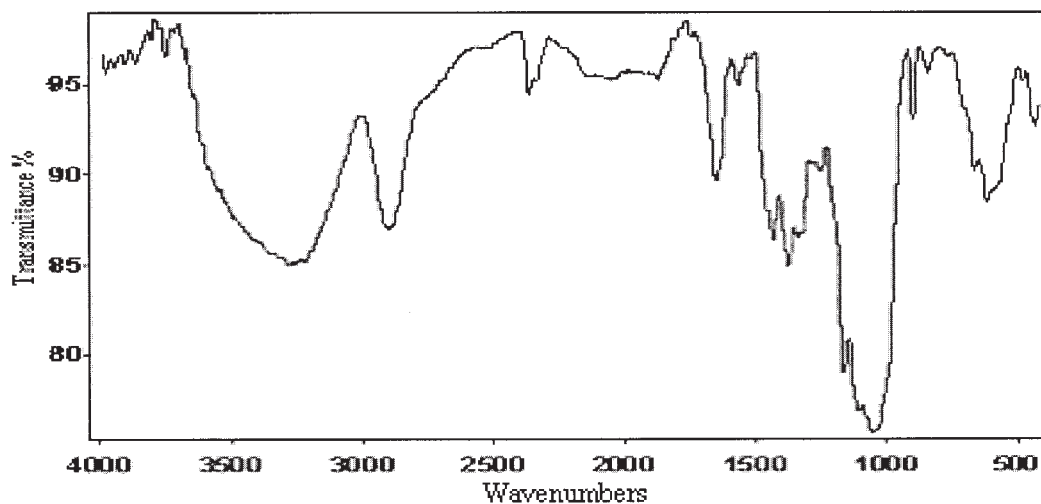
Figure 10 FTIR of wood pulp.

and total irradiation dose. The maize starch was modified by incorporating grafting specific polymers to bring about changes in their physical and chemical properties. Radiation induced grafting of vinyl monomers onto polymeric natural starch, as a convenient way to impart desired properties. Figure 5 represents the effect of irradiation dose on the GE of starch/AAc polymer and Figure 6 represents the effect of monomer percentage on GE. It can be seen that GE increases with irradiation dose, and thereafter, it tends to be a curvature relationship. This is due to the behavior and characteristic of free radical formation in polymers, with irradiation dose, which in general increases at first, then it, tends to level off at higher doses. Meanwhile, the higher the monomer concentration the higher the gel % obtained.

The irradiation dose is an important parameter in any radiation grafting system. In the direct method, the total dose determines the number of the grafting sites. The percentage conversion increases with increasing total dose. An increase in the total dose enhances the formation of radicals in the reaction mixture of monomer, starch, and water. All molecules are activated to induce a higher conversion for both homopolymer formation and the grafting reaction (especially for radiation-sensitive monomers, such as AAc). Increasing the total dose decreases the content of the homopolymer and increases the GE, because the high total dose can induce ample active grafting sites on the starch backbone for the grafting monomer. However, in the case of graft copolymerization of AAc onto starch, at the dose rate of 4.25 kGy/h, when the total dose is higher than 30 kGy for AAc, the homopolymer content increases and GE decreases with increasing total dose. This may be because the higher irradiation dose gives a large amount of radiolysis products (in the absence of oxygen), such as OH and e_{aq}, which initiates homopolymerization rather than the grafting

reaction. At the total dose of 30 kGy for AAc, at a dose rate of 4.24 kGy/h, the radiation modification of starch increased with irradiation dose and monomer concentration.

Surface morphology

The surface characteristics of wood pulp and starch-modified polymer were investigated by SEM and are represented in Figures 7 and 8 to elucidate the topological changes occurring due to grafting reactions and after adsorption of heavy metals. The results do not show the adsorption of heavy metals on the surface of wood pulp and modified starch, and so we used the atomic absorption technique, by using mass spectrophotometer, to investigate the adsorption of metals ions on wood pulp and modified starch.

Characterization of the functional groups of the copolymer by FTIR

The functional groups of the synthesized copolymer were characterized by FTIR. Figure 9 shows that the IR

TABLE I
Maximum Capacity of Metal Uptake (%) After Treatment Time of 8 Days[a]

Metal ion	Adsorption capacity (mg/g)			
	Starch/AAc	Starch/An-amidoxime	Wood pulp	Rice straw
Fe^{3+}	5.75	9.05	6.69	6.75
Cr^{3+}	6.73	8.69	7.41	7.41
Cd^{2+}	2.81	4.16	3.64	2.56
Pb^{2+}	2.06	2.49	6.38	4.51

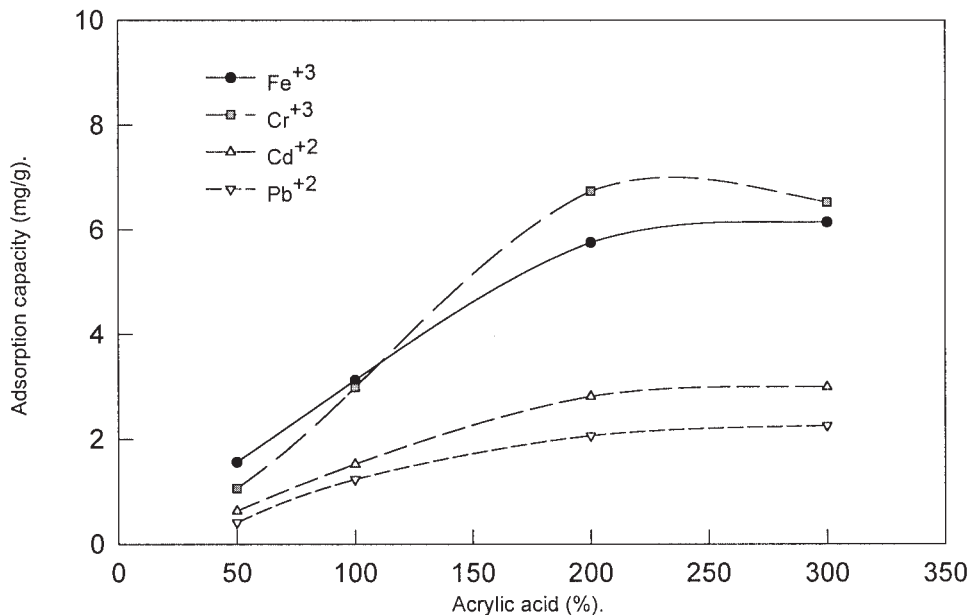[a] Initial concentration of metal ion solution, 100 mg/L; pH, 4.

Figure 11 Effect of AAc monomer:starch on the adsorption capacity (mg/g) of the grafted starch for different metals, at initial metal ion concentration, 100 mg/L; pH, 4; and time, 8 days.

spectra of starch-modified polymer before and after extraction gave all the characteristic absorption peaks of maize starch (3423 cm^{-1} (s, broad); 2927 cm^{-1} (m); 1157, 1082, and 1013 cm^{-1}(s) for the O—H, and C—O stretching, respectively). γ rays produce free radicals at active sites of C2 or C3 hydroxyl of the starch backbones. The grafting reaction emanates from these sites of the swollen starch. For the AAc graft copolymerization, IR spectra before and after extractions gave all the absorption peaks of maize starch and gave the additional peaks at 1718 cm^{-1} (s) and 1259 cm^{-1} (s) of the —COOH group in AAc. Figure 10 shows the FTIR of wood pulp. It was found that the pulped materials have a characteristic broad band, ranging from 3200 to 3600 cm^{-1}, which corresponds to the hydroxyl group of cellulose. The absorption at 2900 cm^{-1} indicates the C—H linkage. Bleached pulps contained carboxylic acid and hydroxyl functional groups.

Removal of some heavy metals by wood pulp and starch/AAc

The factors affecting the affinity of the wood pulp and modified starch, with AAc to remove Pb(II), Cd(II), Fe(III), and Cr(III) from their aqueous solutions, were studied. Also, the study was carried out to determine the appropriate treatment conditions, such as degree of grafting and pH, at which the removal was performed successfully. From Table I, the maximum uptake (%) of metal ions at pH 4 is ordered in the following sequence: $Cr^{3+} > Fe^{3+} >$

$Pb^{2+} > Cd^{2+}$. The initial rate of adsorbed metal ions and the maximum value of uptake are very dependent on the type of metal ions and the steric effect of the copolymer.[36] Also, the ionic size of the investigated metal ions has a great influence not only on its maximum uptake but also on its initial rate. These results may be explained by the difference in atomic radius, ionic radius, valence and ionic electron configuration, and also the nature of the starch/AAc polymer or wood pulp.

TABLE II
Effect of pH on the Adsorption of Various Metal Ions (Fe^{3+}, Cr^{3+}, Cd^{2+}, and Pb^{2+}) on Wood Pulp Starch/AAc[a]

Metal ion	pH	Adsorption capacity (mg/g)	
		Starch/AAc	Wood pulp
Fe^{3+}	2	3.52	3.61
	3	4.52	4.27
	4	5.75	6.69
Cr^{3+}	2	2.47	3.48
	3	3.23	6.71
	4	6.73	7.41
Pb^{2+}	2	1.68	5.79
	3	2.06	6.38
	4	3.08	7.04
Cd^{2+}	2	2.37	2.01
	4	2.81	4.16
	6	3.27	4.61

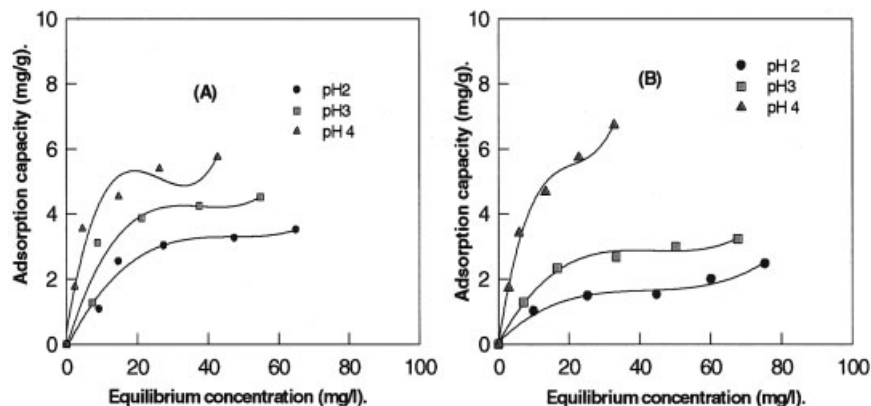[a] Initial metal ion concentration of 100 mg/L; reaction time, 8 days.

Figure 12 Relationship between the equilibrium concentration (mg/L) of metal ions and the adsorption capacity (mg/g) of starch/AAc copolymer at different pH values. (A) Fe^{3+}; (B) Cr^{3+}.

Element	Valence	Atomic radius (Å)	Ionic radius (Å)	Ionic electron configuration
Fe	3+	1.16	0.64	$\{Ar\}^{18}, 4s^2, 3d^6$
Cr	3+	1.17	0.69	$\{Ar\}^{18}, 4s^1, 3d^5$
Cd	2+	1.48	1.03	$\{Kr\}^{36}, 4d^{10}, 5s^2$
Pb	2+	1.75	1.32	$\{Xe\}^{54}, 4f^{14}, 5d^{10}, 6s^2, 6p^2$

This is reasonably understood by considering the diffusion coefficient of these metals through the porous ionic cellulosic materials, which is mainly dependent on their polarity, electronic configuration, ionic radii, *etc.*, and also importantly on the nature of interaction with the functional groups of the cellulosic materials.

Effect of starch: AAc ratio on metal uptake

Figure 11 shows the effect of AAc monomer percentage (%) on the uptake percent of various metal ions through starch/AAc copolymer. It can be seen that the uptake of metal ions increases as AAc amount increases for all metal ions, which may be due to the

increase of the number of functional groups existing in the starch-modified polymer.

Effect of pH of feed solution

The availability of wood pulp and starch/AAc materials for metal-ion complexation was investigated at different pH values in the range from 2 to 4 (for Fe^{3+}, Cr^{3+}, and Pb^{2+}) and the range from 2 to 6 (Cd^{2+}). This is because, at higher pH value, precipitation occurs, which effects the accuracy of the adsorption study. The sorption affinity of the sorbents is listed as a function of pH in Table II.

From the data in Table II and Figures 12–15 it can be concluded that the adsorption of metal ions was remarkably affected by the pH of the solution. Metal ions were adsorbed rapidly from weakly acidic solution and the adsorbed amount increased with the pH value. Modification of adsorption ability of lignocellulosic materials toward metal ions uptake by pulping has been studied by many authors.[37–39] In the metal ion/hydrogel or wood pulp system, metal ion has a cationic character
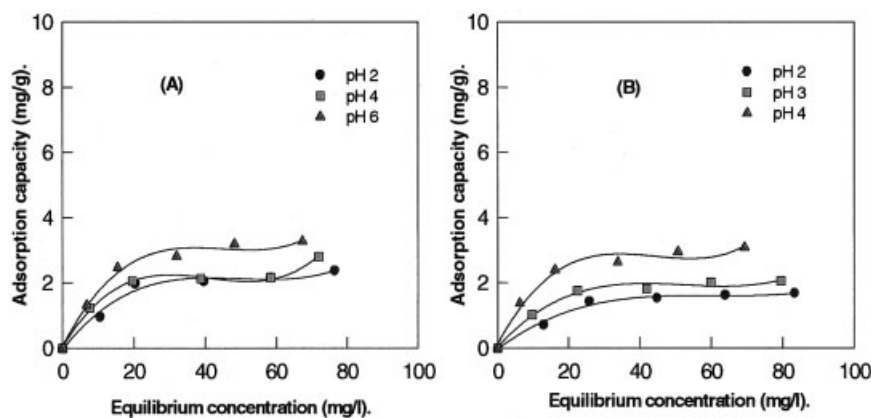

Figure 13 Relationship between the equilibrium concentration (mg/L) of metal ions and the adsorption capacity (mg/g) of starch/AAc copolymer at different pH values. (A) Cd^{2+}; (B) Pb^{2+}.
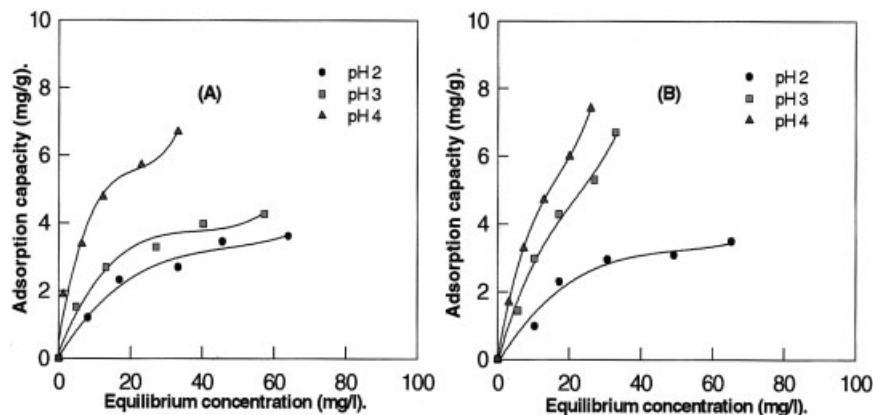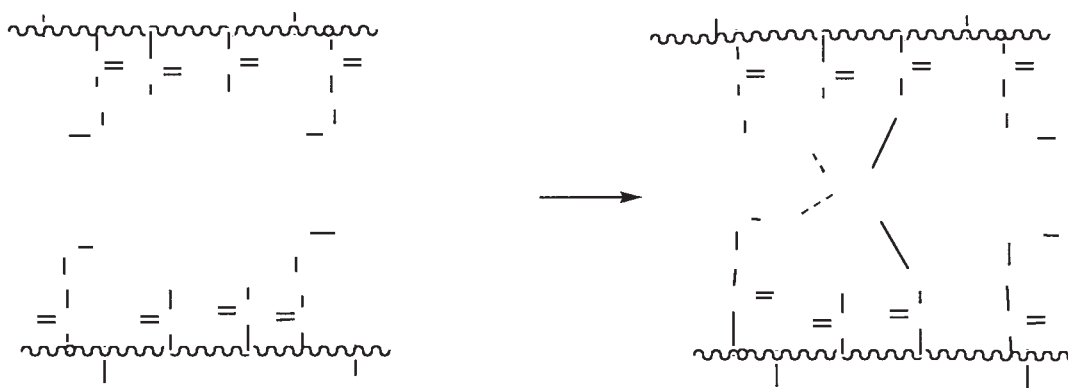
Figure 14 Relationship between the equilibrium concentration (mg/L) of metal ions and the adsorption capacity (mg/g) of wood pulp at different pH values. (A) Fe^{3+}; (B) Cr^{3+}.

and can interact by electrostatic forces with the anionic carboxyl groups of acid in the hydrogel wood pulp.

Possible interactions between metal ion and acidic groups can be represented as follows:[40]

Possible electrostatic interaction between metal ions and starch/AAc copolymer and wood pulp.

CONCLUSIONS

Modification of the macromolecular properties of starch materials by radiation grafting technique, using selected vinyl monomer (AAc), impart new chemical properties, which were proved by physical measurements (FTIR, SEM). The alkaline pulping and bleaching processes have great effect on the adsorption capacity of wood pulp for different pollutants. The results showed that the prepared materials possessed high efficiency in the removal of heavy metals. The
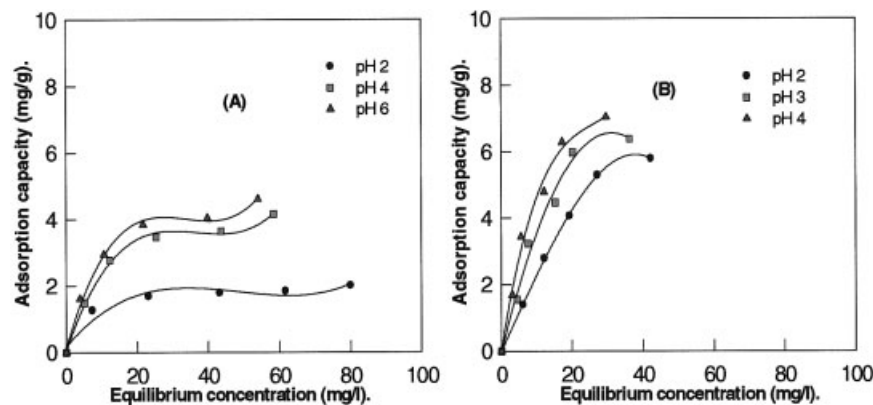

Figure 15 Relationship between the equilibrium concentration (mg/L) of metal ions and the adsorption capacity (mg/g) of wood pulp at different pH values. (A) Cd^{2+}; (B) Pb^{2+}.

change in pollutant adsorption resulting from change in experimental conditions depends on the number and strength of the weak acidic groups present, whereas maximum pollutant uptake depends on the total number of both carboxylic acid and hydroxyl groups present. Also, it should be pointed out that regardless of the substrate used, the value of pollutant uptake differs from one substrate to the other because of the nature of the pollutants, which is essentially governed by the substituents present, steric configuration, and diffusibility.[35]

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