

SYNTHESIS AND CHARACTERIZATION OF GRAFTED CELLULOSE FOR USE IN WATER AND METAL IONS SORPTION

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Graft copolymerization of acrylamide monomer onto cellulose, using ceric ammonium nitrate as initiator, was investigated. Water and metal ions sorption by this grafted cellulose were estimated. The conditions of grafting, e.g. grafting time, dose of initiator, ratio of monomer to cellulose, acid concentration and liquor ratio, were evaluated. The different properties as graft and graft efficiency percentage, as well as polymerization percent, have been determined. Grafted cellulose has been characterized by FTIR and swelling studies. Sorption of different metal ions in the mixture, e.g. Cu, Cr, Ni, and Pb, by grafted cellulose was investigated. The effect of hydrolysis of grafted cellulose by using sodium hydroxide on its swelling properties and metal ions sorption was also investigated. Hydrolysis increases the sorption affinity of grafted cellulose toward water and metal ions.

Keywords: Grafting, Hydrolysis, Selectivity, Swelling, Metal ions uptake, FTIR

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INTRODUCTION

Cellulose is a non-branched polymer of (1→4) β-O-glucopyranose linkage between glucose units. The degree of polymerization of cellulose is affected by the type of treatments. Cellulose is renewable, and due to its abundance in nature it affords material for cost-effective technologies of ion exchange (Nada et al. 2006; Sokker et al. 2006) and hydrogel (Chauhan et al. 2005; Kawabara et al. 1996). Cellulose and its derivatives have been used in metal ion absorption (Murguttim et al. 2000; Nada et al. 2003). Grafting of monomer with hydrophobic and hydrophilic ionic moieties combines a high degree of permeability (Chauhan et al. 2003). Grafted cellulose products, when used as ion exchangers, have advantages over conventional ion exchangers due to chemical resistance, low cost of preparation, and they also offer considerable hydrophilic surface area. Acrylamide and acrylic acid monomers have been successively grafted onto a cellulose backbone (Gitisudhe et al. 2005; Nada et al. 1998). Grafting is a simple technique to incorporate desired active functional groups on the backbone of a polymer for absorption metal ions. Grafting copolymerization of monomers onto cellulose and other natural polymers, e.g. starch and chitosan, supports the metal ions absorption and enzyme immobilization (Chauhan and Lai 2003). Some of the copolymer prepared from cellulose grafting with acrylamide are used for metal ions sorption. Hydrolysis of this grafted cellulose increases its partition coefficient and retention capacity as hydrogel (Chauhan et al. 2003; Chauhan et al. 2005). Cellulose can be derivatized to possess a

number of different functional groups, such as carboxymethyl and amines, to which ions can bind either by chemical or physical adsorption (Nada and El-Wakeel 2006). Grafting copolymerization onto cellulose induces physical changes, since the introduction of side chains leads to different structural characteristics in raw material. Grafting of cellulose shows new properties, such as hydrophobic or hydrophilic character and resistance to chemical and biological agents (Flaquie et al. 2000). Grafting yield can be increased by improving the fiber accessibility to monomer by swelling the substrate before polymerization (Kundu et al. 1998).

The aim of this work is to optimize the graft copolymerization of acrylamide onto cellulose. The effect of hydrolysis of this grafted cellulose on metal ions sorption and swelling percent is investigated. The use of FTIR spectroscopy to follow the molecular structure of the grafted and hydrolyzed grafted cellulose is also investigated.

EXPERIMENTAL

Material

The raw material used in this study was wood pulp (remaining as a waste after manufacture of mesquite mats) at EL Nasr Co. for Intermediate Chemicals, Cairo, Egypt. This material has the following analysis: α -cellulose 86.4, hemicellulose 5.2, and ash content 1 %. The remaining percentage consists of short-chain carbohydrates that are dissolved in water and alkali solution, acrylamide monomer, ceric ammonium nitrate, nitric acid, sodium hydroxide, and a mixture of metal ion solution containing a 20 microgram of each metal (Ni, Cu, Cr, and Pb).

Methods

Graft copolymerization

Grafting was carried out by steeping 5 g of cellulose (w) in acrylamide solution for 5 minutes and then added the dissolved ceric ammonium nitrate in nitric acid at 20°C for 2 hours (total liquor ratio was 25:1, i.e. total quantity of grafting liquor was 125 ml). After grafting, the sample was filtered, washed with distilled water, and then air-dried (w_1). This dried sample was extracted with distilled water in a soxhlet device for 48 hours to dissolve the formed homopolymer. After extraction, the sample was washed with distilled water and then air-dried (w_2). The different variables of the grafted copolymerization of acrylamide onto cellulose were studied e.g. liquor ratio (15:1 – 50:1), nitric acid concentration (0.5 – 0.3 %), initiator concentration (0.075 – 0.225 % based on cellulose), ratio of monomer to cellulose (1:1-4:1), temperature (15–50 °C), and grafting time (0.5-4 hours).

$$\begin{aligned} \text{Polymerization \%} &= [(w_1 - w)/w] \times 100 \\ \text{Graft \%} &= [(w_2 - w)/w] \times 100 \\ \text{Graft efficiency \%} &= [(w_2 - w)/\text{weight of monomer}] \times 100 \end{aligned} \quad (1)$$

Hydrolysis

The grafted cellulose was refluxed with 1.5 % NaOH solution for 2 hours using

a 25:1 liquor ratio. After hydrolysis the sample washed with distilled water till neutralization

Infrared spectroscopy

The infrared spectroscopy was carried out using a KBr disc technique by JASCO FTIR 3006 (Fourier Transform Spectrophotometer). The concentration of each sample was 1 %, the resolution of FTIR instrument was ± 3 , and the baseline was made automatically by the instrument.

Swelling studies

A known weight of grafted cellulose (0.5 g) was swollen by immersion in 50 ml of distilled water at room temperature for 24 hours. The swollen hydrogel was removed, wiped off with tissue paper to remove any superficial water until any water was taken up by new filter paper from the swollen hydrogel, and weighed (w_3).

$$\% \text{ swelling} = [(w_3 - w)/w] \times 100 \quad (2)$$

In another trial different volumes of water (10 -50 ml) were added to constant weight of grafted cellulose, and the swelling percentage was calculated.

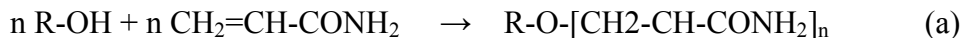
Metal ions sorption

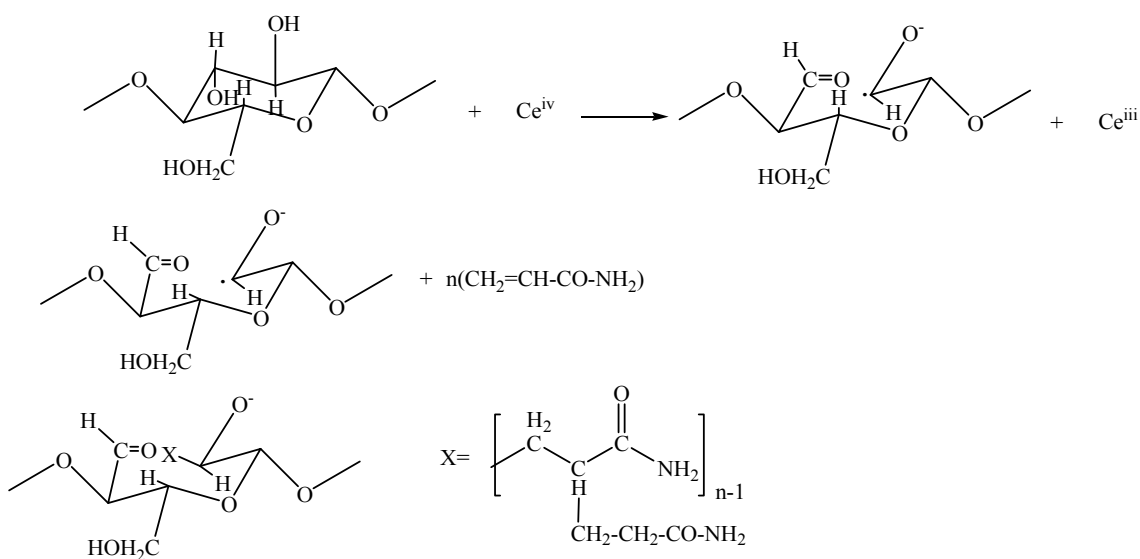
Adsorption of metals was determined by using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). Sorption was carried out by stirring 0.1 g of grafted or hydrolyzed grafted cellulose for 30 minutes in 25 ml solution containing $20 \mu\text{g ml}^{-1}$ ions of (Ni, Cu, Cr and Pb). After filtration, the remaining metal ions in the filtrate were determined using the ICP spectrometer.

RESULTS AND DISCUSSION

Infrared Spectra

Infrared spectra of cellulose, grafted cellulose and hydrolyzed grafted cellulose are shown in Fig. 1. Relative absorbance (ratio of any band intensity / band intensity at 1325 cm^{-1} , which corresponds to CH rocking for cellulose ring) (Yu Levdek et al. 1967) of the OH band at 3420 cm^{-1} was decreased by grafting due to the reaction of acrylamide monomer with the OH of cellulose.





By hydrolysis, the relative absorbance of this band (OH) increased due to the formation of carboxylic groups.

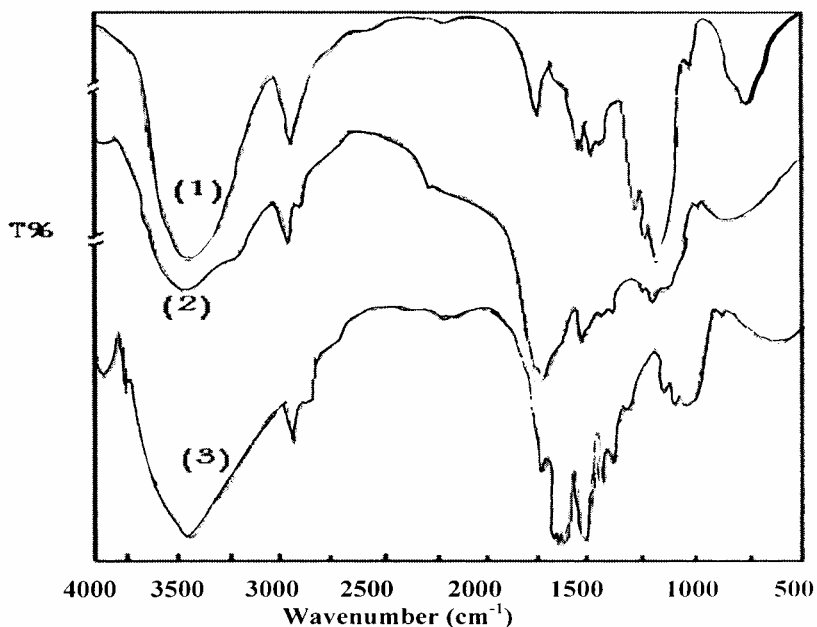
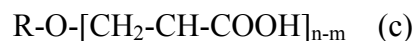
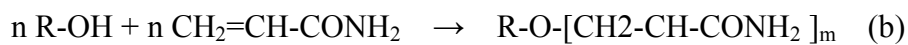


Fig. 1 Infrared spectra of (1) ungrafted, (2) grafted and (3) hydrolyzed grafted wood pulp.

Grafting increased the relative absorbance of CH vibration of CH₂ at 2930 cm⁻¹, as mentioned from the previous equation (a). The ratio of this band intensity to band intensity of the OH group at 3420 cm⁻¹ had a higher value in grafted than that in case of ungrafted wood pulp (Table 1). The relative absorbance of the band at 1650 cm⁻¹, which is characteristic to C=O of the amide group, increased by grafting, and it decreased by hydrolysis due to the change of some CONH₂ groups to carboxylic groups, which appeared at 1715 cm⁻¹. The ratio of the band intensity at wavenumber 1650 cm⁻¹ to the band intensity at 1115 cm⁻¹ (ether linkage between glucose units in cellulose chains) increased by grafting due to the increase of incorporated CONH₂ group onto cellulose. On the other hand, the relative absorbance of C-O-C band at 1115 cm⁻¹ decreased by grafting and hydrolysis. This means that some degradation of cellulose occurred during grafting and hydrolysis. The crystallinity index (ratio of band intensity at 1425 cm⁻¹ / band intensity at 900 cm⁻¹) (Nelson et al 1964) of cellulose was decreased by grafting due to the incorporation of amide groups. By hydrolysis, this crystallinity index was decreased due to the swelling of crystalline regions

Table 1. The Relative Absorbance of Some Bands in Wood Pulp, Grafted and Hydrolyzed Grafted Wood Pulp

Wavenumber cm ⁻¹	Group	Relative absorbance		
		w.p.	Grafted w.p	Hydrolyzed-grafted w. p.
3419	OH	1.92	0.95	1.45
2920	CH ₂	1.02	1.27	1.20
2820	CH ₂	-----	0.72	0.091
1650	CONH ₂	0.82	2.38	1.39
1715	COOH	---	-----	1.48
1114	C-O-C	1.74	0.99	0.90
Ratio 1654 / 1120	CONH ₂ /C-O-C	0.38	1.38	1.19
Ratio 2920 / 3419	CH ₂ /OH	0.54	0.72	0.69
1425 / 900	Cr.l.	2.43	2.1	1.96

Effect of Different Variables on Grafting Process

Nitric acid concentration

Figure 2 shows the effect of nitric acid concentration on the grafting parameters (polymerization, graft and graft efficiency %). These parameters were increased by increasing acid concentration from 0.5 to 1 % (Fig. 2). Increasing the acid concentration more than 1 % decreased these parameters.

The explanation of such behavior is that nitric acid in the grafting medium assists the grafting by causing inter- and intra-crystalline swelling of cellulose and also acting as a catalyst in hydrolysis of cellulose, leading to an improvement in monomer accessibility. Also 1% acid concentration is sufficient to establish a balance between the suppression of the rate of the active species formation and the rate of primary radicals. On the other hand, the experiment showed that the homopolymer formation was also enhanced in the

presence of acid. A higher concentration of acid, however may cause a degradation of backbone chains of cellulose as well as graft chains. Moreover, high acid concentration causes an oxidation of the formed free radicals, which decrease the rate of initiation with an increase of the homopolymer.

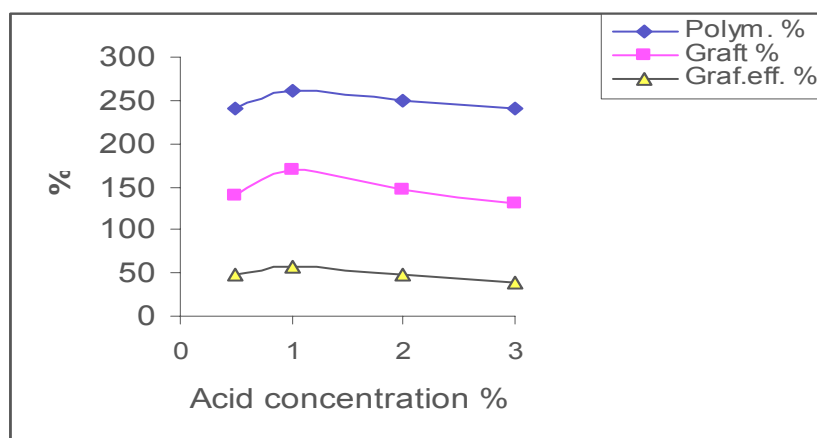


Fig. 2. Effect of acid concentration on the grafting parameters
L.R. 25:1, initiator conc. 0.15%, monomer to cellulose 3:1, Temp. 20°C for 2 hrs.

Ratio of monomer to cellulose

Figure 3 shows the effect of ratio of acrylamide to wood pulp on the different parameters of grafting. It is clear from the figure that the increase of ratio of monomer to cellulose increased the graft and graft efficiency %.

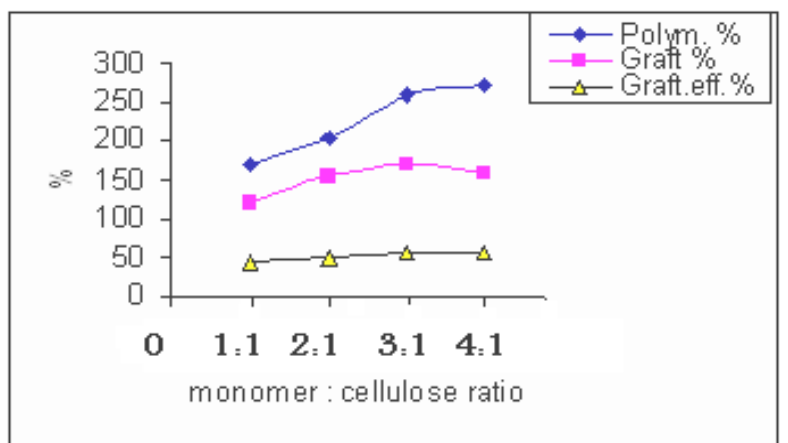


Fig. 3. Effect of monomer to cellulose ratio on the grafting parameters
L.R. 25:1, initiator conc. 0.15%, acid conc. 1 %, Temp. 20°C for 2 hrs

Enhancement in graft % can be attributed to the immobility of cellulose macroradicals. Hence, the availability of acrylamide molecule in the proximity of these radicals is a prerequisite for graft initiation and propagation. The enhancement can be due

to the formation of long graft chains. Increasing the ratio of monomer to cellulose more than 3:1 causes a slightly increase in graft % with increase the homopolymer formation due to the increase in the viscosity of grafting solution, which decreases the penetration rate of monomer solution through the cellulose chains, causing an increase of homopolymer formation.

Effect of initiator

The effect of initiator concentration on the grafting process is shown in Fig. 4. It can be observed that the graft and graft efficiency percentage were increased with increasing initiator concentration up to 0.15 %. This increase resulted due to the formation of a great number of grafting sites on the cellulose backbone, which, in presence of monomer, induced grafting. By increasing the initiator concentration more than 0.15 %, the graft and graft efficiency were slightly decreased, but the polymerization percentage was slightly increased. The increase of initiator more than 0.15 % caused a retardation of monomer diffusion through cellulose chains, accelerating the rate of termination. On the other hand, the increase in the initiator leads to initiation of many chains, which enhances the chance of mutual initiation of growing polymeric chains. These factors increase the formation of homopolymer and cause a fall in graft yield (Sikdar et al. 2003)

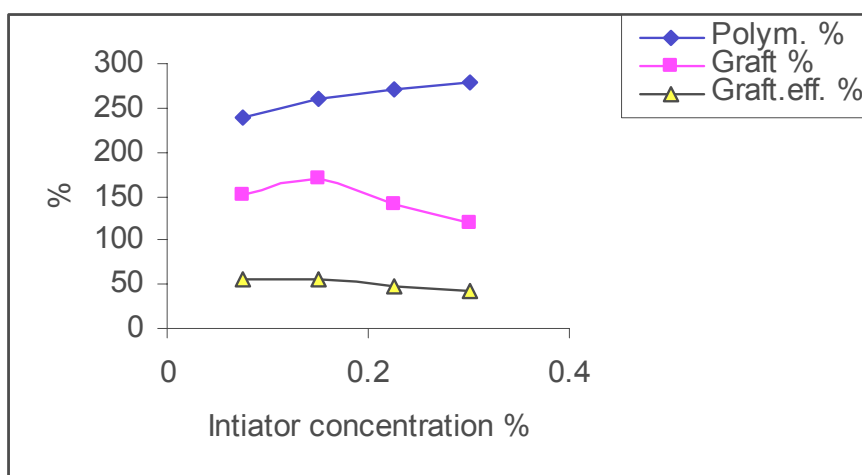


Fig. 4. Effect of initiator concentration on grafting parameters
L.R. 25:1, monomer to cellulose ratio 3 : 1, acid conc. 1 %, Temp. 20°C
for 2 hrs.

Effect of liquor ratio

It was found that an increase of liquor ratio up to 25:1 caused an increase in graft and polymerization percentage (Fig. 5). This can be explained by the increase of dispersion of cellulose in the grafting liquor, causing an increase in the contact between cellulose and monomer.

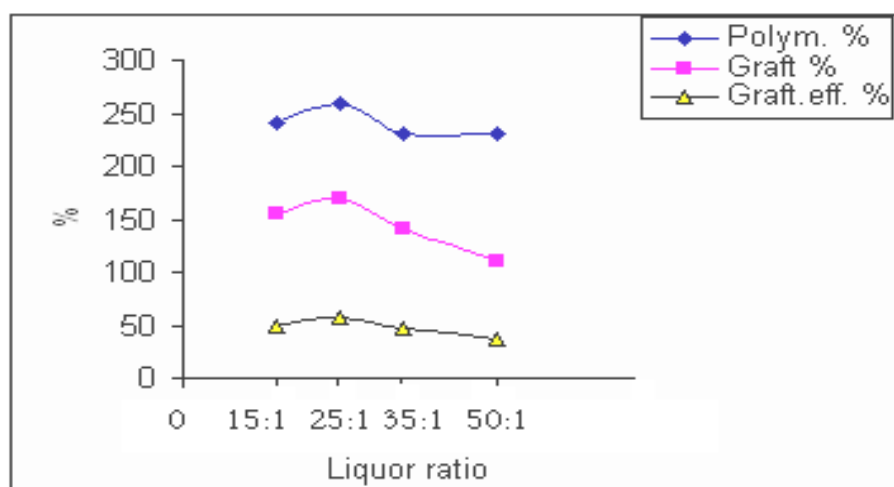


Fig. 5. Effect of liquor ratio on grafting parameters; Initiator concentration 0.15 %, monomer to cellulose ratio 3 : 1, acid conc. 1 %, temp. 20°C for 2 hrs.

Increasing the liquor ratio more than 25:1 decreased the graft and graft efficiency percentage, while a slight increase in the polymerization percentage was obtained. This can be due to the dilution of monomer and chemicals, which decreases from the contact between chemicals and cellulose.

Reaction time

Figure 6 shows the effect of reaction time on different grafting parameters. It can be seen from the figure that the increase of reaction time from 0.5 to 2 hours increased the grafting, graft efficiency, and polymerization percentage. Increasing the reaction time more than 2 hours caused the reaction rate to level off. This leveling off can be ascribed to the depletion in monomer and initiator, as well as the shortage of available grafting sites as the reaction proceeds. So, the graft and graft efficiency percentage increased with increasing reaction time to 2 hours. This increase in graft after particular time is considered to be a detrimental effect.

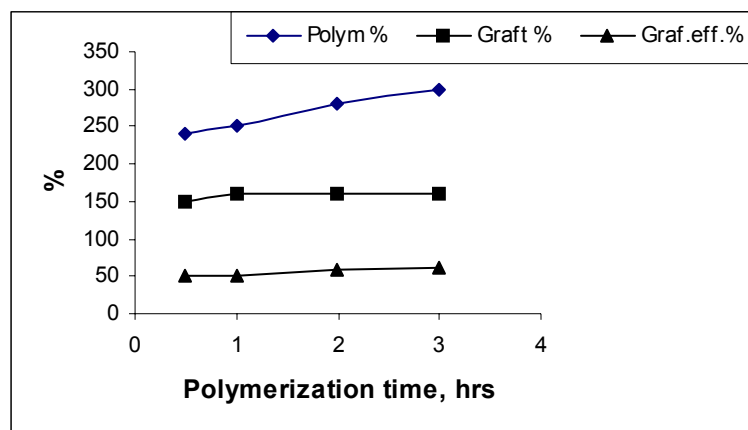


Fig. 6 Effect of time on the grafting parameters; Initiator concentration 0.15 %, monomer to cellulose ratio 3 : 1, Acid conc. 1 % Liquor ratio 25 : 1 Temp. 20°C

Reaction temperature

Graft copolymerization of acrylamide onto cellulose increased by increasing the reaction temperature from 15 to 20 °C, as shown in Fig. 7.

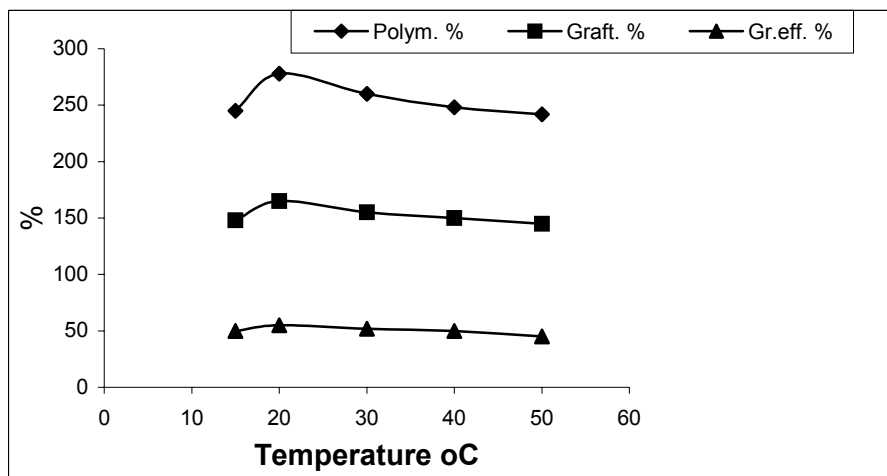


Fig. 7. Effect of reaction temperature on grafting parameters; Initiator concentration 0.15 %, monomer to cellulose ratio 3 : 1, acid conc. 1 % ,Liquor ratio 25 : 1 reaction time 2 hours

This effect is attributed to the increase of the rate of diffusion of monomers through cellulose chains, swelling of cellulose, as well as an increase of the rate of initiation and propagation of the grafting process (Nada and Youssef 1997). Increasing temperature more than 20 °C enhances the oxidation of free radicals, and mutual termination of growing macroradicals favors more homopolymer formation over the grafting reaction.

Treatment of Pulp

Before the grafting process, the wood pulp was treated with acid (sulphuric acid) at 1.5 % for 2 hours or with alkali soda at 3 % for 1 hour under reflux. Figure 8 shows the different grafting parameters of untreated, acid-, and alkali-treated wood pulp. The grafting of treated pulp with acid had higher grafting parameters than alkali- and untreated wood pulp. This can be attributed to the fact that acid treatment causes a decrease in the degree of polymerization (DP 580), and consequently the end groups of cellulose chains, causing an enhancement in the reactivity of the wood pulp toward grafting. On the other hand, the grafting of alkali-treated wood pulp (DP 910) was higher than that in case of untreated wood pulp (DP 890). This was due to the increase in the penetration rate of grafting chemicals through the cellulose chains because of the decrease in the crystalline parts of the alkali-treated wood pulp. The DP (degree of polymerization) of treated pulp with alkali is higher than untreated pulp due to the dissolving of small chains of cellulose during alkali treatment.

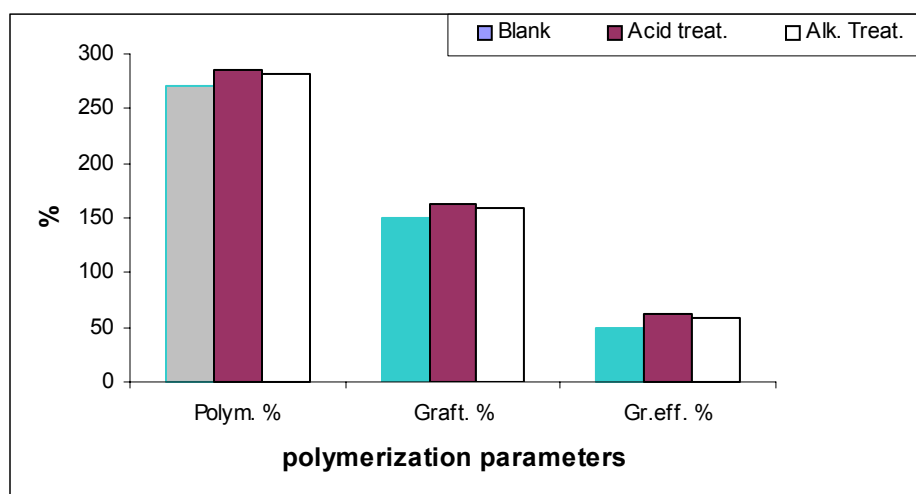


Fig. 8. Effect of different treatment of wood pulp on the Grafting parameters

Water Absorption

Table 2. Water Absorption (g/g) and Swelling (%) of Grafted and Hydrolyzed Grafted Wood Pulp

Samples	Graft %	Water uptake g/g		Swelling %	
		Graft.	Hydroly.	Graft.	Hydroly.
Ungrafted w. P.	0	1	1.8	61	104
Grafted untreated pulp	170	12.7	80.2	1080	7920
Grafted Acid. Treat. W.P.	190	11	54	1100	5000
Grafted alkal.treat.W.P.	180	11.8	85.2	1200	8600

Condition of grafting : L. R. 25 : 1, acid concentration 1 %, monomer : wood pulp ratio 3:1, initiator concentration 0.15 % (based on wood pulp), reaction temperature 20°C for 2 hours

The swelling percentage and water uptake were measured by steeping 0.5 g of sample in distilled water for 24 hours. The surface water on swollen samples (grafted and hydrolyzed grafted wood pulp) was removed by safely pressing between the folds of filter paper. Table 2 illustrates the swelling percentage and water uptake g/g. From the table it is clear that the grafted and hydrolyzed grafted acid-treated wood pulp had lower values of water uptake and swelling percentage than corresponding tests in case of untreated and alkali-treated wood pulp, although it had a higher graft percentage than the other pulps. This may be due to the higher proportion of crystalline regions of acid treated wood pulp (Table 1). This increase in the crystalline regions in the grafted, acid-treated wood pulp decreased the rate of diffusion of water through the grafted cellulose chains. On the other hand, water uptake and swelling percentage of the grafted wood pulp was higher than that in case of non-grafted wood pulp. This can be explained by the opening up of the matrix of wood pulp due to considerable loss of crystalline regions, which increased the diffusion of water through the grafted cellulose chains. Grafted alkali-treated wood pulp had a higher swelling percentage and water uptake than the grafted untreated pulp. This was due to the decrease in crystalline regions of grafted alkali treated wood pulp, which increased the diffusion of water through grafted cellulose

chains. Moreover, the hydrolyzed, grafted samples had a higher swelling percentage and water uptake than the grafted samples (Table 2). This is explained mainly by the change of some amide groups, which are incorporated in grafted samples to carboxylic groups.

So, the hydrolyzed grafted samples contained two different hydrophilic functional groups (-COOH, -CONH₂), which increased the swelling and water uptake for hydrolyzed samples. To show the effect of volume of water on swelling percentage and water uptake, known weights of hydrolyzed grafted samples (0.25 g) were steeped in different volumes of water for 24 hours (Table 3). It is seen from table that the swelling percentage increased with increasing the volume of added water to the hydrolyzed grafted wood pulp. This was due to the difficulty in dispersing the substrate in low volume of water, which decreased the diffusion rate of water through the substrate. Increasing the volume of added water enhanced the movement of substrate in water, and consequently the diffusion of water through cellulose chains increased.

Table 3. Effect of Volume of Water on the Swelling % and Water Uptake of Hydrolyzed Grafted Untreated, Acid and Alkali Treated Wood Pulp

Samples	Water added	10	20	30	40	50
		Swelling %				
Hydrolyzed grafted untreated w. p.		940	4700	6600	8000	8720
Hydrolyzed grafted acid treated w.p.		896	4400	6000	7630	8400
Hydrolyzed grafted alkali treated w.p.		903	4500	6050	7900	8500

Metal ions uptake

In this part, 0.1 g of the grafted or hydrolyzed grafted wood pulp was steeped with stirring in 25 ml of solution containing 20 µg ml⁻¹ of different metal ions (Cu, Cr, Ni, and Pb) for one hour. After steeping, the sample in solution was filtered, and the remaining metal ions in the filtrate were determined. Table 4 shows the metal ions uptake and retention capacity by grafted pulp.

Table 4. Metal ions Uptake and Retention Capacity of Grafted and Hydrolyzed Graftd Wood Pulp

Samples	Metal ions uptake %				Retention capacity			
	Cr	Pb	Mn	Ni	Cr	Pb	Mn	Ni
Grafted wood pulp	94.5	90	92.5	82	189	180	185	165
Hydrolyzed grafted wood pulp	97	92	95	90	194	185	190	180
Ungrafted wood pulp	8	4	3	1.5	16	8	6	3

$$\text{Metal ions uptake \%} = \frac{\text{amount of metal in polymer}}{\text{amount of the metal in the feed}} \times 100$$

$$\text{Retention capacity} = \frac{\text{amount of metal ions in polymer}}{\text{weight dry of polymer}}$$

Structural aspects of the polymeric backbone are important factors affecting metal ions sorption. The prepared grafted cellulose copolymers remove metal ions both by adsorption on N of amide groups and also by sorption in the bulk of grafted copolymer hydrogel. Therefore, the structure of a polymeric hydrogel affects the level of polymer interaction with water and the provision of active sites to absorb or coordinate metal ions. Hence, the sorption behavior and the quantity of metal ions taken up depend, in addition to the attributes of metal ions, also on different structural aspects of the polymer. It is clear from Table 3 that the metal ions uptake percentage and retention capacity of Cr were higher than for Mn, Cu, and Ni. This can be attributed to the fact that the Cr ion has a lower atomic radius than other metal ions and consequently its adsorption by polymer is high. In general, the amount of metal ions uptake by ion exchanger is affected by the electronegativity and hydrated values of metal ions. The sequence of metal ions sorption was as follows: Cr > Mn > Pb > Ni. Hydrolysis of the hydrogel affected the metal ions uptake percentage and retention capacity by activation of functional groups of grafting chains by opening up the hydrogel network and activating amide groups into more active groups, such as carboxyl groups, which have a stronger tendency to chelate and exchange ions (Couhan et al 2005). Partial hydrolysis of amide groups increased water uptake of hydrogel and increased the tendency of functionalized hydrogel, leading to enhanced metal ions sorption. So from Table 4 it is seen that the metal ions uptake percentage and retention capacity of hydrolyzed, grafted wood pulp was higher than for grafted wood pulp.

CONCLUSIONS

1. The optimum conditions of graft copolymerization of acrylamide onto wood pulp were liquor ratio 25:1, ratio of monomer to wood pulp 3:1, nitric acid concentration 1 %, and initiator concentration 0.15 % (based on wood pulp) at 20 °C for 2 hours.
2. Grafted acid-treated wood pulp had a higher graft and graft efficiency percentage, as well as polymerization percentage than grafted, untreated and alkali-treated wood pulp.
3. Water absorption and swelling percentage of the grafted, alkali-treated wood pulp were higher than corresponding results in case of grafted untreated and acid treated wood pulp.
4. Hydrolysis of grafted alkali treated wood pulp increased its affinity toward water absorption and swelling percentage. Also it improved its affinity toward metal ions, increasing the metal ion uptake and retention capacity.

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