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# Green Modification of Waste Cotton Surface Fabric by (2-acrylamide-2-methyl Propane Sulfonic Acid, AMPS) and Nano Magnetite Particles

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A GREEN polymer containing specific functional groups with the ability to absorb high dosages of contaminated dyes & heavy metals from the waste water was achieved. The surface properties of the waste cotton tissues were improved through its surface modification by graft polymerization using γ-radiation $^{60}$Co to acquire new properties such as hydrophilic or hydrophobic character based on the chemical structure of the used monomer. The nucleophiles surface cotton fiber modification was done by introducing an anionic monomer (2-acrylamide-2-methyl propane sulfonic acid, AMPS) and nano magnetite particles to easily facilitate separation of toxic substances. Factors influencing the grafting such as monomer concentration, radiation dose were studied. The structure surface properties of the grafted fabric were characterized by XRD, SEM, TGA and IR. The results obtained show high and green removal efficiency of Cd(II), Co(II) and cationic dyes (methylene blue) from waste water.

**Keywords:** Waste cotton, Cationic dyes, Graft polymerization, γ-radiation $^{60}$Co, Nano magnetite particles.

## Introduction

Industrial wastes containing hazardous compounds such as toxic heavy metals Cu, Cd, Co and synthetic dyes which are a major pollutant mainly from dyeing textiles, pulp manufacturing, paper, etc, are hardly biodegradable due to their complex aromatic structures [1,2]. The treatments of dye-containing effluents and toxic heavy metals were done previously by various traditional approaches such as chemical precipitation, ion exchange, complications and photo degradation [3-6]. Recently, adsorption is an efficient technique which is greatly used for the removal of industrial contaminants due to its economic viability, low cost and environmentally safe [7]. Several kinds of natural polymers as adsorbents have been tested, such as luffa cylindrical [8, 9], sugar cane bagasse [10-13], wheat straw [14-17], jute fiber [18], kapok [19], starch [20], rice husk [21], etc. Most of these materials have drawbacks in adsorption selectivity due to the surface charge diversity of pollutants. Hence, functionalization based on grafting on its surfaces is expected as a promising adsorbent owing to its adsorption capability for cationic pollutants. Cellulose is the most abundant renewable polymer on earth. It is a linear polysaccharide consists of β-d-glucopyranose units joined by β-1,4-glycosidic linkages (Fig. 1). Various attractive chemical and physical properties for cellulose like hydrophilicity, stereoregularity, presence of reactive hydroxyl groups, biocompatibility, biodegradability, and ability form supra structures [22, 23]. The major sources of cellulose are cotton, wood and aerobic bacteria. The point of helpfulness of cellulose could be developed by its surface functionalization through graft polymerization. Different polymerization methods have been utilized to get cellulose-based graft copolymers from cellulose and its derivatives with an incredible assortment of monomers. These strategies are generally characterized into three main groups: (1) chemical grafting, (2) plasma-induced grafting, and (3) radiation-induced grafting. Radiation-induced graft polymerization is a green direct and easy technique for the modification of virgin polymers yielding new properties with super-efficient behaviors [24-26].

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Three kinds of radiations that have been generally utilized are γ-radiation, electron beam, and UV-radiation. Cotton is the most abundant renewable biopolymer having specific properties such as excellent hydrophilicity, hollow flat banded structure, biocompatibility and biodegradability. Up to 40% of cotton grown is wasted between the harvest and the manufacturing of garments and textiles. The aim of this study based on green functionalization of waste cotton fibers by graft polymerization using γ-radiation proceeds through free-radical mechanism. These free radicals initiate the graft polymerization of 2-acrylamide-2-methyl propane sulfonic acid (AMPS) monomer on cotton surface (Fig. 2). The purpose behind picking waste cotton fiber as supporting materials of adsorbents is that the main constituent cellulose, which can be modified or functionalized easily by introducing various functional groups or nanoparticles to hydroxyl surface moiety. The present study also describes the adsorption of Co(II) , Cd(II) and cationic dyes from aqueous solution by graft polymerization of cotton fabric with AMPS and nano magnetite particles, which produce high surface fabric with small particle size distribution having anionic character.

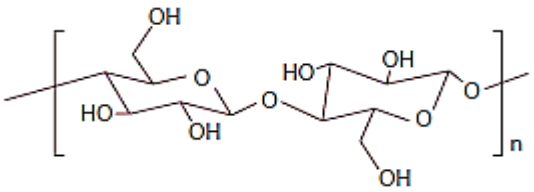


**Fig. 1. Structure of cellulose.**

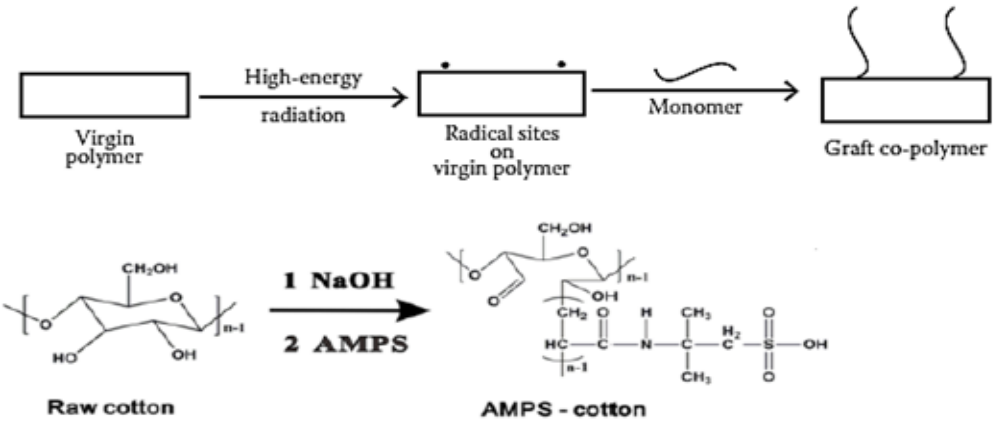


**Fig. 2. Graft polymerization of raw cotton fabric with AMPS under high-energy radiation.**

## Experimental

*Materials and Methods*

2-Acrylamido-2-methyl-1-propanesulfonic acid (AMPS) monomer, Nano magnetite particles prepared previously [27], (cationic dye) (methylene blue), Cadmium nitrate tetrahydrate $Cd(NO_3)_2.4H_2O$, Cobalt(II) nitrate hexahydrate $Co(NO_3)_2 \cdot 6H_2O$ . All chemicals used in the present study were purchased from Sigma Aldrich and used as such without any further purification. Waste cotton fibers are collected from harvest and the manufacturing of garments and textiles.

*Pretreatment of Cotton Fibers*

8 g cotton fibers were refluxed in 200 ml boiling NaOH aqueous solution (2 wt%) for 24 hr. Then the fibers were filtered and transferred into 150 ml NaOH aqueous solution (18 wt%), and the mixture was stirred at room temperature for 2 h. Then the fibers were filtered, and washed for three times with sufficiently of deionized water. After drying at 60 °C for 2 hr, the motivated cotton fiber was obtained.

*γ-irradiation (green polymerization) for grafting AMPS onto cotton surface fabrics*

Various contents of AMPS 15-65 wt%

dissolved in N, N dimethylformamide (DMF) were added to fixed weight of the treated waste cotton fabric located in flat flask. The graft polymerization were done using $^{60}$Co gamma source at different radiation doses (10-60) kGy at two dose rates 1.46kGy/h and 3.9kGy/h. The grafted fabrics were washed several times with DMF, and then washed with hot water for 8 h in order to extract any untreated monomer.

*γ-irradiation (green polymerization) for preparation magnetite nanoparticles loaded on grafted AMPS/cotton*

A definite weight of the treated waste cotton fabric was placed in a flat bottomed flask, an appropriate amount of AMPS 15-65 wt% dissolved in N,N dimethylformamide (DMF) and (0.5-2) gm of nano magnetite particles dispersed in DMF were added and the polymerization reaction was allowed to proceed using $^{60}$Co gamma source at different radiation doses(10-60) kGy at two dose rates 1.46kGy/h and 3.9kGy/h. The grafted fabrics and nanofabrics were washed several times with DMF, and then washed with hot water for 8 h in order to extract any homopolymer that may have attached to the surface of the fabrics. The grafted sample was then allowed to dry in an air oven at 70$^{o}$C until constant weight was reached (figure 3). The grafted percentage was determined by the percent increase in weight as follows:

$$\% \text{ Graft} = [(w_g - w_0)/W_0] *100$$

where $w_0$ and $w_g$ represent the weights of initial and grafted fabric, respectively.

*Characterizations of treated cotton surface*

The FTIR spectrum of treated cotton surface has been recorded by Mattson 1000, Pye-Unicam spectrometer, in the range 400–4000 cm$^{-1}$ for elucidating the structure. Shimadzu TGA system, TGA-50 H, was used to illustrate the thermal stability of treated cotton surfaces. SEM imaging of the grafted and chemically modified cellulosic fabric was obtained on a JSM-5600L at 20 kV. The specimens were prepared for SEM by freeze fracturing in liquid nitrogen and applying a gold coating of approximately 300 Å. A single beam U.V.visible spectrometer, Milton Roy spectronic 1201, U.S.A was used to perform accurate and reliable measurements. Atomic Absorption Instrument Shimadzu, AA-6300, using hallow cathode lamps for the determination of Co and Cd. Merck atomic absorption standard solutions for the selected metals were used for calibration.

*Equilibrium degree of swelling*

The clean, dried, grafted sample of known weights (1gm) was immersed in distilled water at room temperature for various time intervals reaching maximum or equilibrium at 48 h, then each interval removed from water and the excess water on the surface was discarded, blotted by absorbent paper and quickly weighed. The equilibrium degree of swelling (EDS) of the prepared grafted fiber was calculated as

$$EDS = (Ws - Wi)/(Wi) g/g$$

where Ws is the weight of the grafted cotton fiber at equilibrium and Wi is the weight of the dried grafted cotton fiber before swelling.

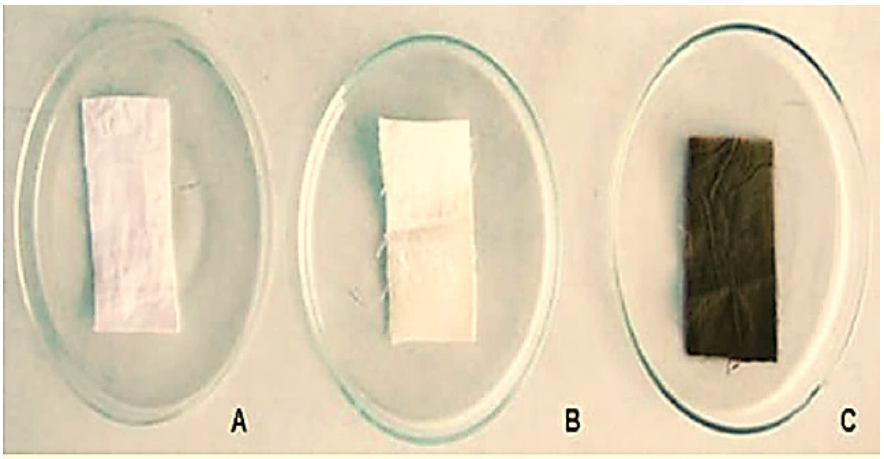


**Fig. 3. Photograph showing (A) cotton cellulose fabric (B) grafting AMPS/cotton and (C) Magnetite nanoparticles loaded on grafted AMPS/cotton fabric.**

*Gel fraction*

Gel fraction was characterized for cotton cellulose fabric and magnetite nanoparticles loaded on grafted AMPS/cotton fabric samples as follows. A known weight of the sample (Wo) was extracted in a refluxing system by boiling in bidstilled water for 6 hours. The samples were then removed and dried in ambient air to get rid of excess water to reach a constant weight (W1). Finally, the soluble fraction was calculated according to the following equation:

$$\text{Soluble fraction \%} = [(Wo - W1) / Wo] \times 100$$

Where: $W_0$: initial weight of the dry sample, W1: the weight dry insoluble gelled part after extraction,

$$\text{Gel fraction (\%)} = 100 - \text{soluble fraction (\%)}.$$

Where: W1: initial weight of the sample, W2: final weight of the swelled sample.

*Swelling behavior (Water uptake %)*

A known weight of the sample was soaked in distilled water for different time intervals at room temperature; the sample was then removed and blotted on filter paper to remove the excess water on the surface. The water uptake % was calculated using the following equation:

$$\text{Water uptake \%} = [(W2 - W1) / W1] \times 100$$

Where, W1: initial weight of the sample, W2: final weight of swollen sample.

*Evaluations of treated cotton surface for water purifications*

*Dye sorption measurements*

The sorption of methylene blue dye by untreated and treated cotton cellulose fabrics with magnetite nanoparticles or grafted AMPS monomer were carried out by a general procedure based on spectrophotometric measurements of the dye solution before and after dye sorption. In this procedure, a certain concentration from each dye was dissolved in boiled water without any additives. Subsequently, a constant weight of sample was then immersed in dye solution at different pH values and the dye uptake was determined by measuring the light absorption of the residual dye solution. The dye sorption by samples was determined according to the following equation:

Dye uptake% = Dye concn. / initial dye concn. X 100

The light absorption measurements were performed using a UV/ Vis spectro photometer made by Unicam England.

*Sorption of heavy metal ions*

Untreated and treated cotton cellulose fabrics with magnetite nanoparticles or grafted AMPS monomer were used to remove some heavy metals such as $Co^{2+}$ and $Cd^{2+}$ from wastewater. Various solutions of mineral salt ($Cd(NO_3)_2.4H_2O$ and $Co(NO_3)_2.6H_2O$) were prepared from the known concentrations (250 ppm) for the first time. Then a sample of the known weight (1.5 g) of the material prepared in these solutions was immersed for different periods of time ranging from one hour to 24 hours. The percentage of metal absorption (%) was determined using the following equation:

$$\text{metal uptake \%} = [(C_o - C_1) / C_o] \times 100$$

where C1 and $C_o$ are the concentrations of the metal solution before and after sorption by magnetite nanoparticles loaded on grafted AMPS/cotton fabric, respectively.

## Results and Discussion

*Grafting*

Figure 4 showed the Effect of AMPS concentrations on degree of grafting of AMPS onto cotton fiber irradiated at 40 kGy at two dose rates 1.46 kGy/h and 3.9 kGy/h. Figure 5 showed the effect of irradiation dose on the degree of grafting of AMPS monomer concentration (45%) onto cotton fiber at various irradiation doses (10-60) kGy at two dose rates 1.46 kGy/h and 3.9 kGy/h. It was found that the grafting efficiency increased with increasing AMPS concentration and with increasing irradiation doses reached maximum at 40 kGy. This is may be due to reactivity effect of the structure of AMPS vinyl monomers increased by irradiation dose and monomer concentrations therefore enhanced grafting process. Figure 6 explained the effect of magnetite nanoparticles concentration on the degree of grafting of AMPS monomer (45%) onto cotton fiber irradiated at 40 kGy at two dose rates 1.46 kGy/h and 3.9 kGy/h.It was clear from Figure 6 that, grafting increased by increasing nanoparticles concentration reaching maximum at 8wt%. This might be due to an increase of nano particles leading to increase surface area of cotton fabrics and thus increasing the chance of functionalization or grafting of nano/AMPS onto cotton surface.

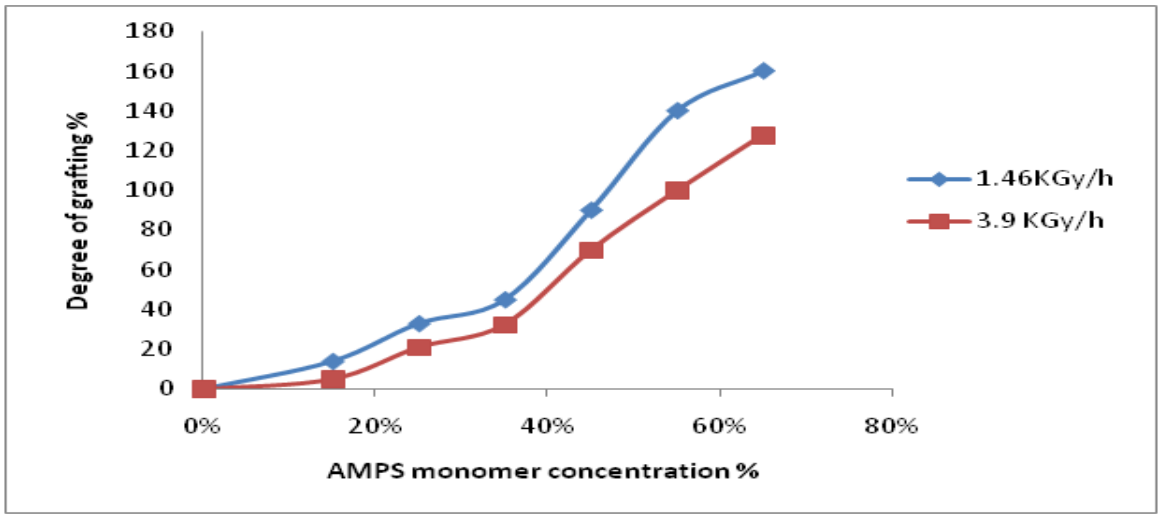


**Fig. 4. Effect of AMPS concentration on degree of grafting of AMPS onto cotton fiber irradiated at 40 kGy at two dose rates 1.46 kGy/h and 3.9 kGy/h.**

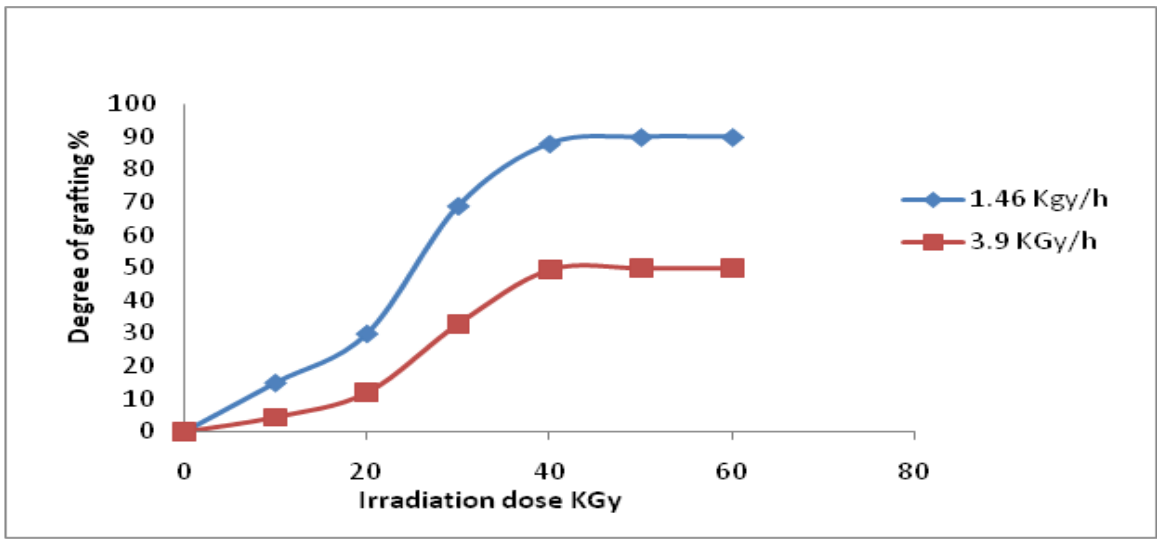


**Fig.5. Effect of irradiation dose on the degree of grafting of AMPS monomer concentration (45%) onto cotton fiber at different irradiation dose rate.**

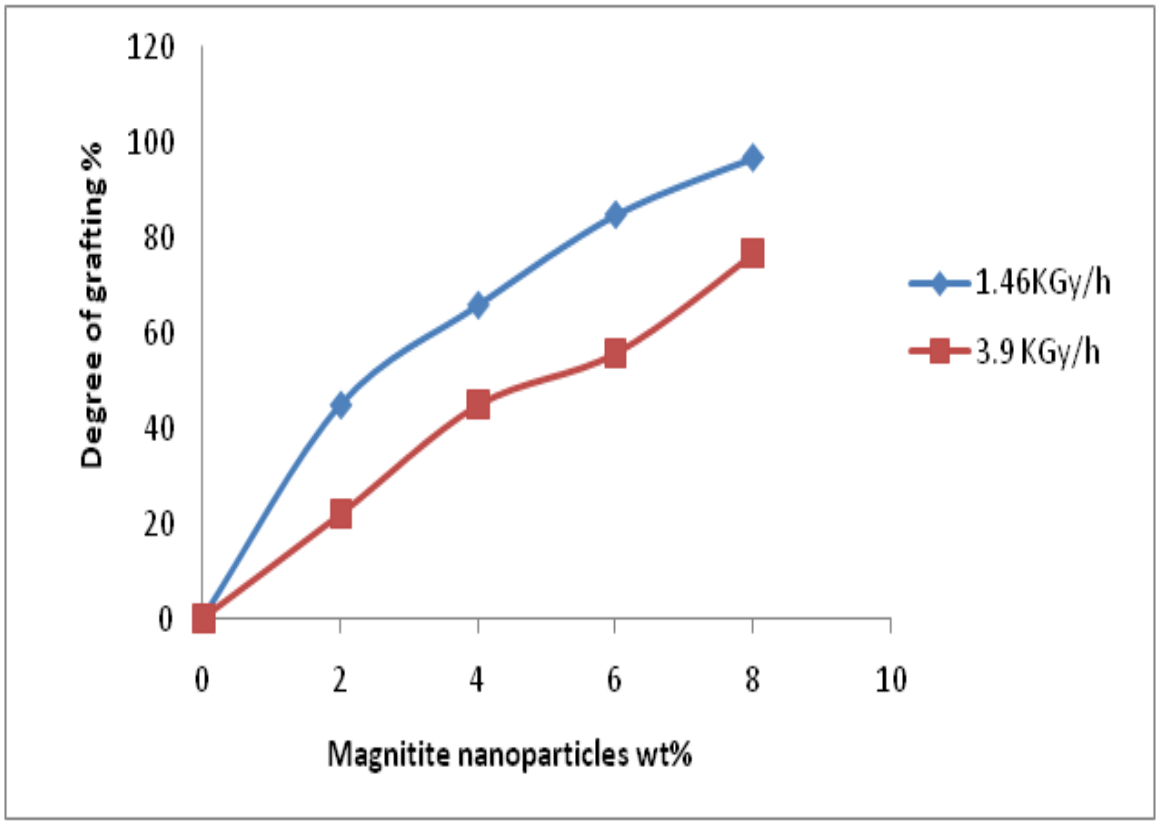


**Fig.6. Effect of magnetite nanoparticles concentration on the degree of grafting of AMPS monomer (45%) onto cotton fiber irradiated at 40kGy using two dose rates 1.46 kGy/h and 3.9 kGy/h respectively.**

*Gel fraction*

Figure 7 represented the effect of irradiation dose (kGy) on the gel fraction % on treated cotton cellulose fabrics with magnetite nanoparticles (8wt%) and various AMPS monomer concentrations at various irradiation doses (kGy) at different concentrations of AMPS monomers. It is clear from the figure that, gel fraction increases with increasing AMPS content till 45% and then decreased. This may be due to high reactivity of AMPS monomer which means that all monomers grafted with cotton matrix but by increasing AMPS monomer after 45% the gel fraction decrease this may be attributed to steric effect AMPS hinders the grafting reactions. On the other hand gel fraction increase with increasing irradiation dose till 40 kGy then decreased. For a given polymer, high irradiation dose typically effects on its mechanical and physical properties. The magnitude of detrimental dose rate effects will differ based on macromolecular structure, formulation, and environmental conditions. Increasing irradiation dose more than 40 kGy may crack the formed grafted cross-linked cotton matrix so the gel fraction decreases.

*Swelling behavior*

Figure 8 represented the equilibrium degree of swelling behavior of Magnetite nanoparticles loaded on grafted AMPS/cotton fabric at different AMPS composition and various irradiation doses (10-60) kGy at two dose rates 1.46kGy/h and 3.9kGy/h respectively, which calculated by equation (2) as a function of time as shown in the experimental section. It was found that the swelling behavior of the prepared modified cotton samples increased with increasing AMPS concentration up to 45 wt% using 8wt% nano particles and 40 kGy. This is may be due to increase the concentration of hydrophilic carboxylic groups of AMPS. While as irradiation dose increase the swelling behavior decreased. This is may be due to the increase the crosslinking density, which resulted in narrowing of the pore size and reduces the free spaces available to water retention [28, 29].

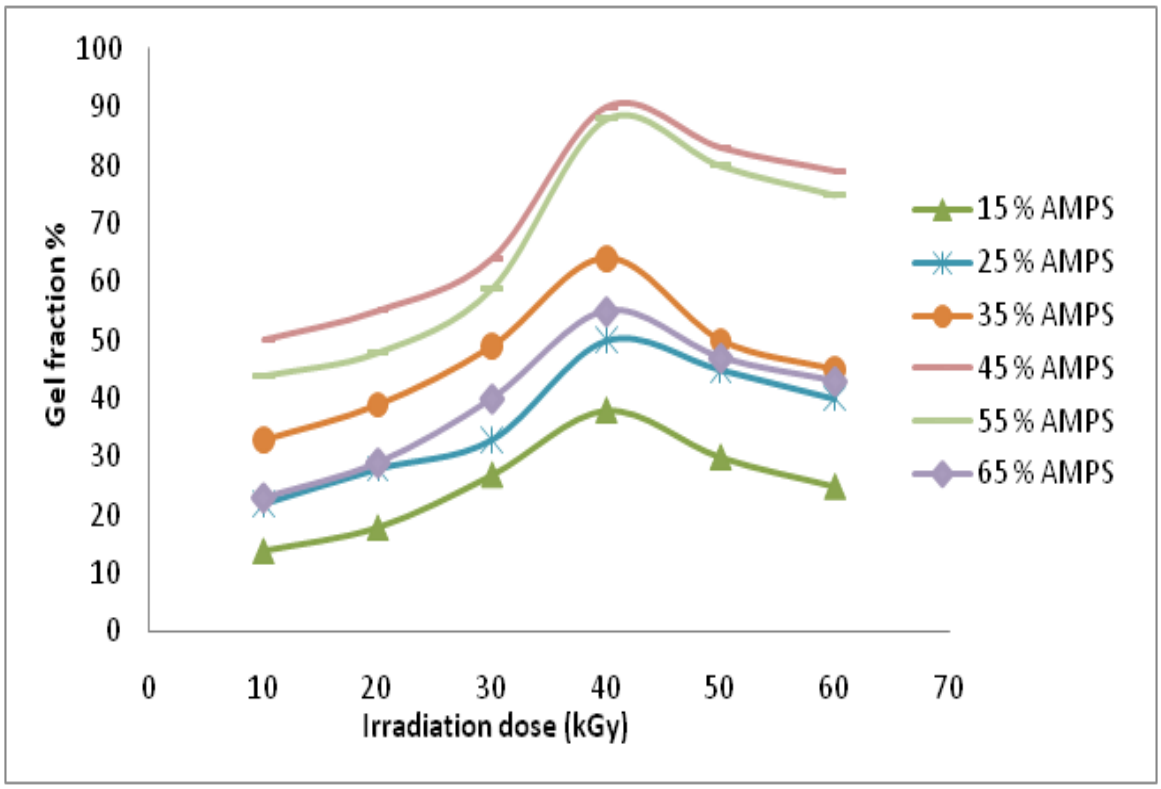


**Fig.7. Effect of irradiation dose (kGy) on the gel fraction % on treated cotton cellulose fabrics with magnetite nanoparticles (8wt%) and various AMPS monomer concentrations.**

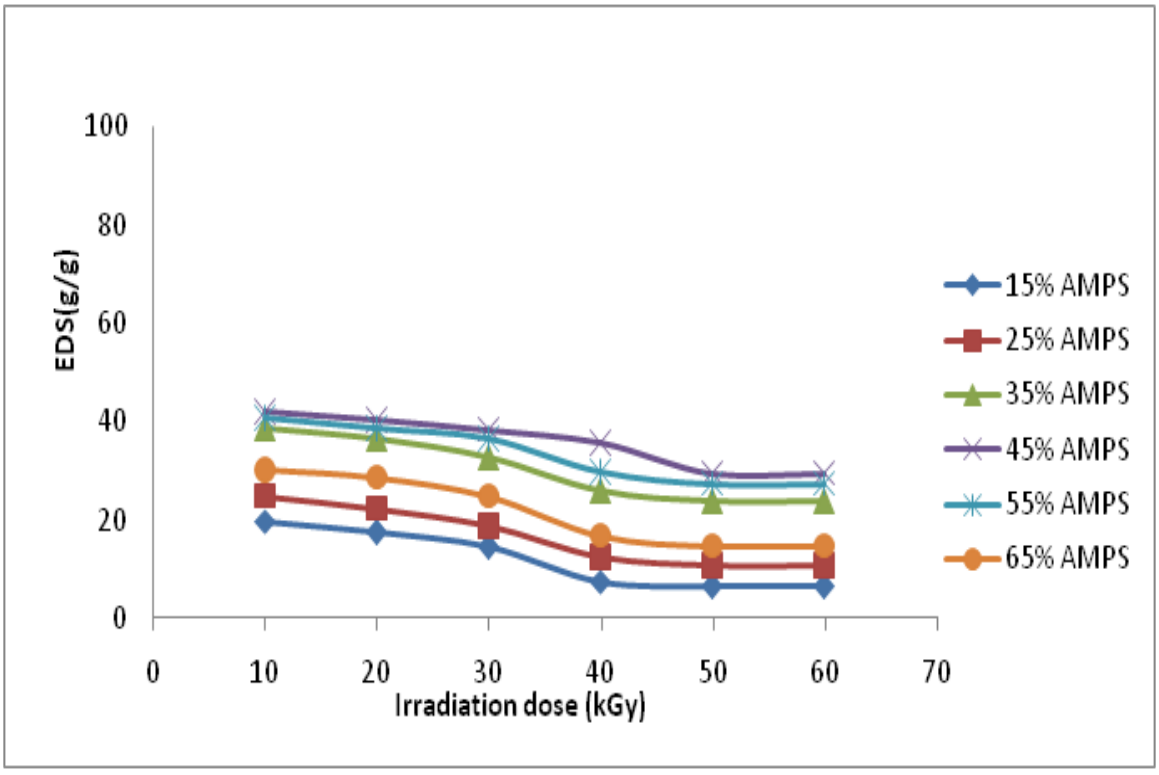


**Fig. 8. Effect of absorbed irradiation dose (kGy) on the equilibrium degree of swelling (EDS) (g/g) of cotton–AMPS grafting at different AMPS concentration.**

Infrared spectroscopy was carried out to illustrate the chemical structure of the prepared treated cotton matrixes. Figure 9 (a, b, c) showed comparative interpretation of FTIR spectra of magnetite nanoparticles, untreated cotton and the prepared magnetite nanoparticles 8wt% loaded on grafted AMPS 45wt%/cotton fabric samples at 40 kGy respectively. In case of Fig. 9(b) it showed a broad absorption band between 3100 and 3700 $cm^{-1}$ centered around 3360 $cm^{-1}$ illustrated characteristics of OH functional groups of cotton matrix cellulose. With grafting process (Fig. 9c) this band showed a decrease in intensity indicating a gradual increasing of the extent of grafting degree. A strong adsorption band with a maximum at 1030 $cm^{-1}$ is a result of the overlapping bands attributed to functional groups of cellulose, namely the C–C, C–O and C–O–C stretching vibrations. The same intensity increase is seen in this band, too suggesting that the cotton matrix were occupied with magnetite nanoparticles. A strong absorption peak around 450 $cm^{-1}$ on nano deposited cotton fabrics' FTIR spectra can be attributed to nanoparticles as seen in Fig. 9(c) [30]. And a peak of the –CO carbonyl group is observed at 1651$cm^{-1}$. Asymmetric C-H stretching is obtained in the 2000–2500 $cm^{-1}$ range, whereas the symmetric peak is obtained in the 2600–2800 $cm^{-1}$ range. A broad peak at 3400–3600 $cm^{-1}$ in the spectrum of the grafted sample is due to the presence of carboxylic groups of AMPS. In addition, the peak at 3282 $cm^{-1}$, corresponding to carboxyl groups functionalities in grafted sample, is shifted to 3340 $cm^{-1}$ in Fig. 9(c). This also indicates the binding of magnetite particle with oxygen in carboxyl groups [31].

TGA and the rate of thermal decomposition were considered the used methods to illustrate the thermal stability of polymers over a wide range of temperature. The thermo grams of magnetite nanoparticles, untreated cotton and the prepared magnetite nanoparticles (8wt% loaded on grafted AMPS 45wt%/cotton fabric samples) at 40 kGy respectively were shown in Figure 10 (a, b &c).

Two further small weight losses from 250 to 800 $^0$C were observed in the untreated cotton may be attributed to the decomposition of the cotton fibers. In case of use of magnetite nanoparticles (8wt %) loaded on grafted AMPS (45wt %) on cotton sample had lower water content loss and a better thermal stability than the untreated fiber from 350 to 800 $^0$C compared to the thermal stability of the first decomposition for pure cotton. In contrast, the TGA of pure cotton fiber reveals substantial weight loss in three stages. In the first stage, from 50 to 250 $^0$C, the weight loss is due to moisture loss. Cotton fibers are hydrophilic in nature, so one can expect some water content to be retained by the cotton fibers. In the second stage, from 250 to 370 $^0$C, the cotton fiber displays a great weight loss, which is attributed to the degradation of cellulose, as expected from the literature [32]. In the third stage, in the temperature range of 370–800 $^0$C, the fiber continues to decompose slowly with 10 % of the weight retained. It could be seen that nano grafted AMPS on cotton increase thermal stability from 250 to 350 % in first composition and from 400 to 550 compared with untreated cotton fiber in the last composition. Therefore, the grafting was acting to protect cellulose from the effect of thermal degradation introducing barrier to reduce the entrance of air and the resultant oxidative degradation. Accordingly, thermal degradation resistance of cotton fiber could be improved by the use of grafting. X-ray diffraction of the magnetite nanoparticles and the prepared magnetite nanoparticles (8wt %) loaded on grafted AMPS (45wt %) on to cotton fabric sample at 40 kGy were performed and showed in Figure 11 (a, b, c) respectively. Comparing X-ray diffraction of the magnetite nanoparticles with that of the original cotton or grafted one to clarify their crystalline structure and the Change caused by the grafted modification. The crystalline characteristics of magnetite nanoparticles (8wt %) loaded were clearly observed at (15-45) 2θ values. The presence of these peaks confirms the formation of magnetite nanoparticles on the surface of cotton fabric. On the other hand, diffraction peaks intensities of crystallite of grafted cotton decreased compared to magnetite nanoparticles intensity. Similar results of XRD analysis were reported in literature for cotton modified sample [33].

Scanning electron microscope is the best tool for morphological analyses studies [34]. The scan electron microscope images of original cotton, magnetite nanoparticles (8wt %) loaded on grafted AMPS (25, 35 or 45wt%)/cotton fabric samples at 40 kGy were shown in Figure 12(a, b, c ,d). Figure 12(a) showed different magnifications of pure cotton. The morphologic changes of cotton after modification could be clearly observed from these images (Fig. 12 (b, c & d). Cotton modified with various concentrations of AMPS and magnetite nanoparticles images cleared the presence of variable amounts of foreign deposited materials on their surfaces. Based on the SEM results, it was reasonable to assume that the presence of these deposits might be responsible for the variation of cotton property resulting from distribution of

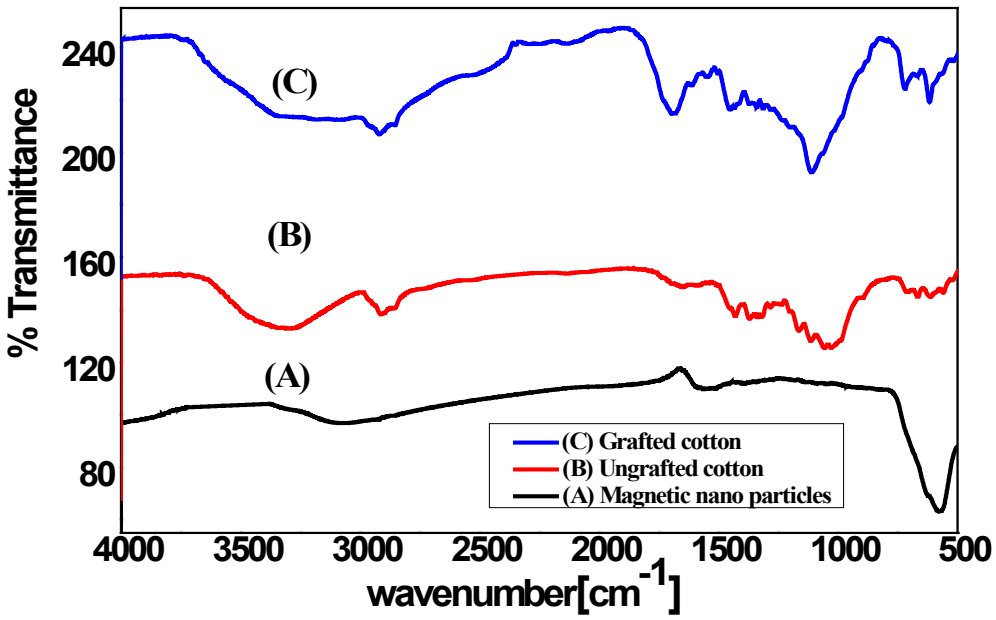


**Fig. 9 (a, b, c). FTIR spectra of magnetite nanoparticles, untreated cotton and the prepared magnetite nanoparticles (8wt% loaded on grafted AMPS 45wt%/cotton fabric samples at 40 kGy respectively.**

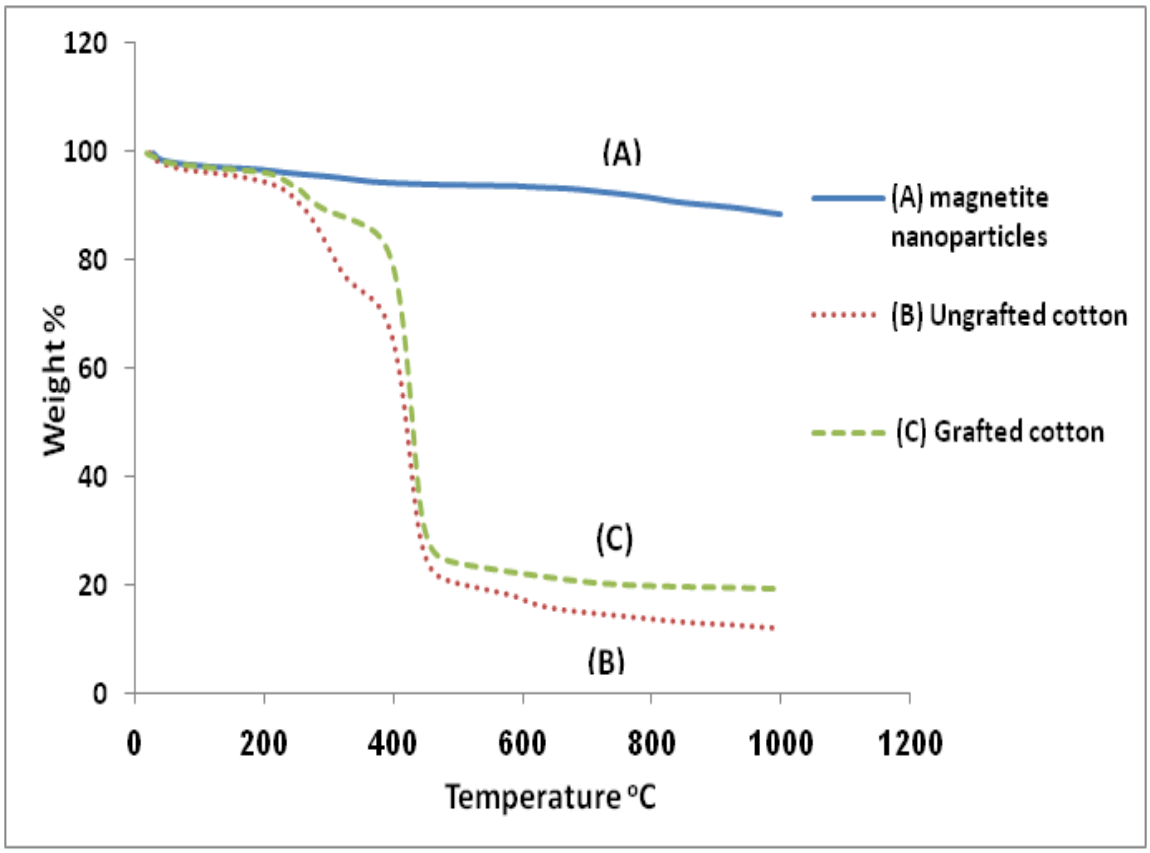


**Fig. 10 (a,b,c). The thermogram of magnetite nanoparticles, cotton and the prepared magnetite nanoparticles (8wt% loaded on grafted AMPS 45wt%/cotton fabric samples at 40 kGy respectively.**

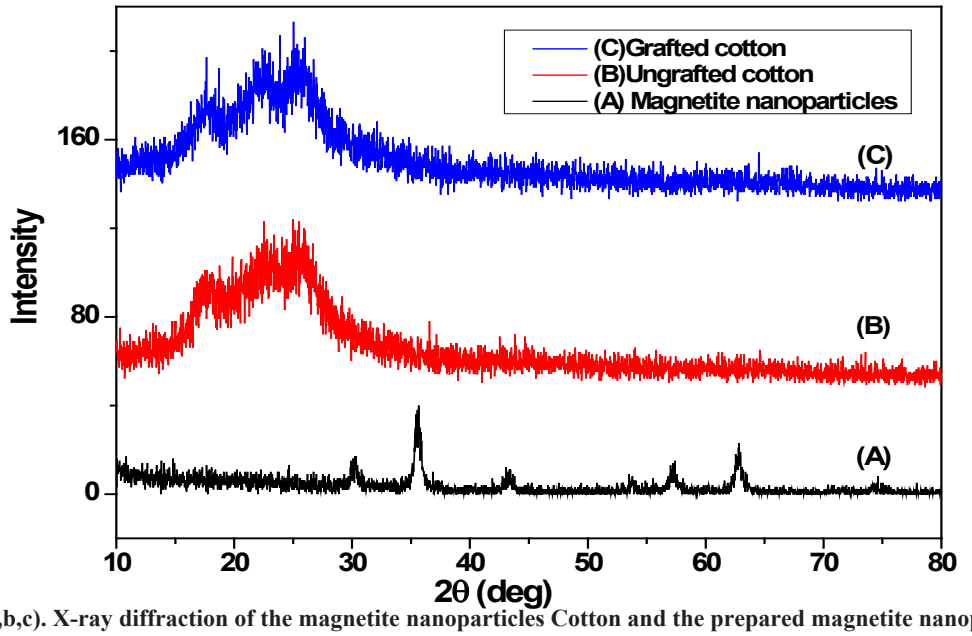


**Fig. 11 (a,b,c). X-ray diffraction of the magnetite nanoparticles Cotton and the prepared magnetite nanoparticles (8wt %) loaded on grafted AMPS (45wt %) cotton fabric sample at 40 kGy respectively.**

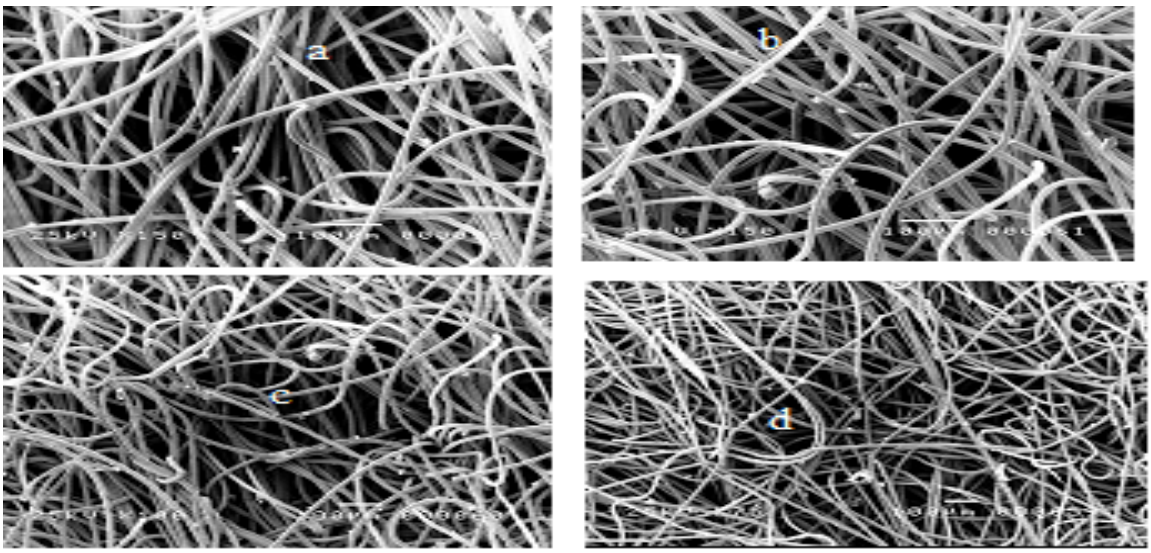


**Fig. 12 (a, b, c ,d). SEM images of a) original cotton b) magnetite nanoparticles (8wt%) loaded on grafted AMPS (25wt%)/cotton fabric, c) magnetite nanoparticles (8wt%) loaded on grafted AMPS (35wt%)/ cotton fabric, d) magnetite nanoparticles (8wt%)loaded on grafted AMPS (45wt%)/cotton fabric, samples at 40 kGy respectively.**

nanoparticle and grafting AMPS monomer over all cotton surfaces.

*Wastewater Treatment*

The modified waste cotton with various compositions of AMPS and various contents of magnetite nanoparticles grafted at dose 40 kGy were selected due to its high swelling behavior and high thermal stability and used for application in removal of different pollutants (metal ions and dyes from wastewater prepared in lab).

*Treatment of waste water from metal ions*

There are several methods for removing metal ions from aqueous solution, such as chemical precipitation, membrane filtration ion exchange, and adsorption on the grafting [35-40]. The adsorption capacities of the prepared modified

grafting waste cotton with AMPS monomer and magnetite nanoparticles toward copper $Cd^{+2}$ and cobalt ions $Co^{+2}$ were evaluated. Figure 13 showed the effect of nano magnetite % grafting cotton–AMPS on the adsorption capacities of Co (II) and Cd (II) uptake at various pH values. The adsorption capacities increased rapidly with the increase of percentage of nanoparticle reaching maximum at 8%. It was found also that, the equilibrium adsorption capacities of the tested grafted nano cotton fiber reached to 90 % adsorption. Figure 13 also showed the effect of pH solution on the sorption of Co (II) and Cd (II) ions was studied. The pH ranges from 2–6 had been chosen carefully to match the term with the industrial wastewater (2.0–5.5 for Co (II) and Cd (II) ions) [41]. Metal ion removal studies at pH > 6 were not conducted because of the precipitation of $Cd(OH)_2$ or $Co(OH)_2$ from the solution. One can see that the removal efficiency increased with the increase in pH for both Cd and Co ion reaching maximum at pH 5.5 for both ions. On the other word, the higher the acidity, the lower the ion adsorption; this might be due to the protonation of the –COOH group in the acrylic part under acidic conditions, generating repulsive charges between these functional groups and metal ions [42]. It was clear that the adsorption efficiency for Cd ion is more than that of Co ions. Due to large ionic radius of $Cd^{+2}$ ions than that of $Co^{+2}$ where the maximum value of metal ions uptake is very dependent on the ionic size of the investigated metal ions and the steric effect of the structure of grafted cotton.

*Factors affecting on Removal of cobalt and cadmium ions from aqueous Solution*

*Effect of AMPS concentration*

The effect of AMPS concentration on metal ions removal efficiency of grafting waste cotton with various concentrations of AMPS from 15 to 65% monomer and 8% magnetite nanoparticles were determined as shown in Figure 14. It was clear from the figure that, metal ions uptake increased by increasing AMPS content reached the maximum uptake at 45% AMPS content for all samples. This content might attributed increase the adsorption of tested ions on the active sites of the side chains protruded from the main cotton chains (adsorption process) i.e increasing surface area for adsorption. The data also revealed that the carboxylic group was more efficient in binding metals than the hydroxylic group and amine group [43] thus the amounts of metal ions removed from the aqueous solutions increased with increasing the amount of AMPS substrate. The data also reveal that the selectivity toward Cd(II) ion was higher than that toward Co(II) ion at the same conditions which shows the selectivity of the prepared samples toward specific metal. This selectivity increased with increasing of the carboxyl group content of polymers due to the spontaneous tendency for salt formation. The greater atomic size of Cd(II) than cobalt makes the salts of Cd(II) more stable than those of cobalt. The obtained data agree with those of the corresponding literature [42, 43].

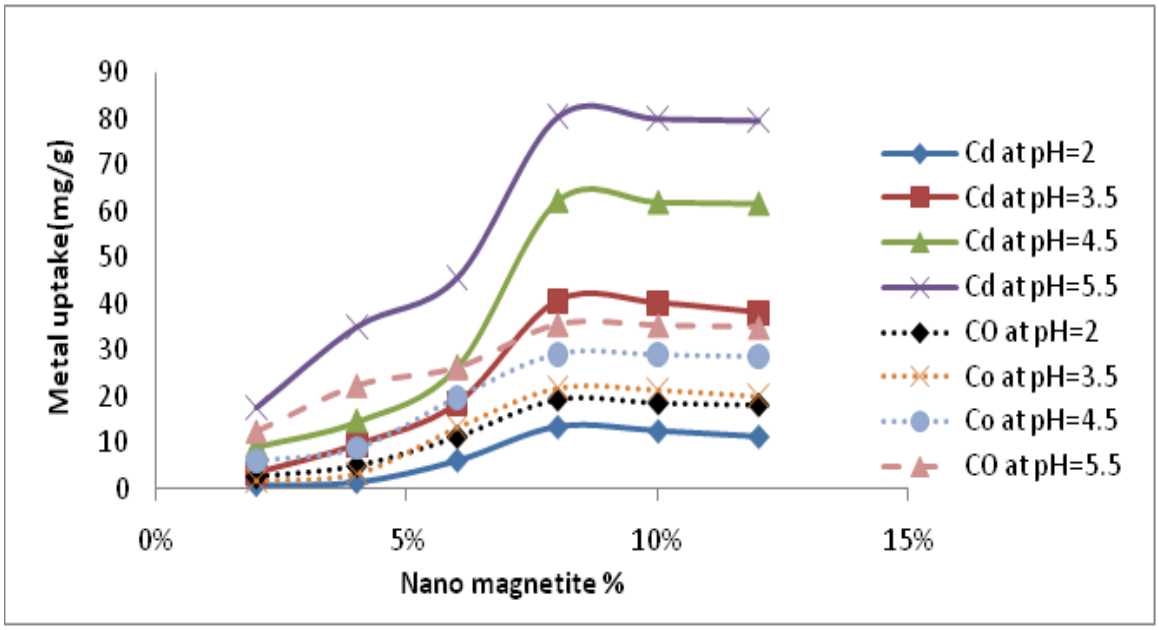


**Fig. 13. Effect of Nano magnetite % in grafting cotton–AMPS on Co (II) and Cd (II) uptake at various pH values.**

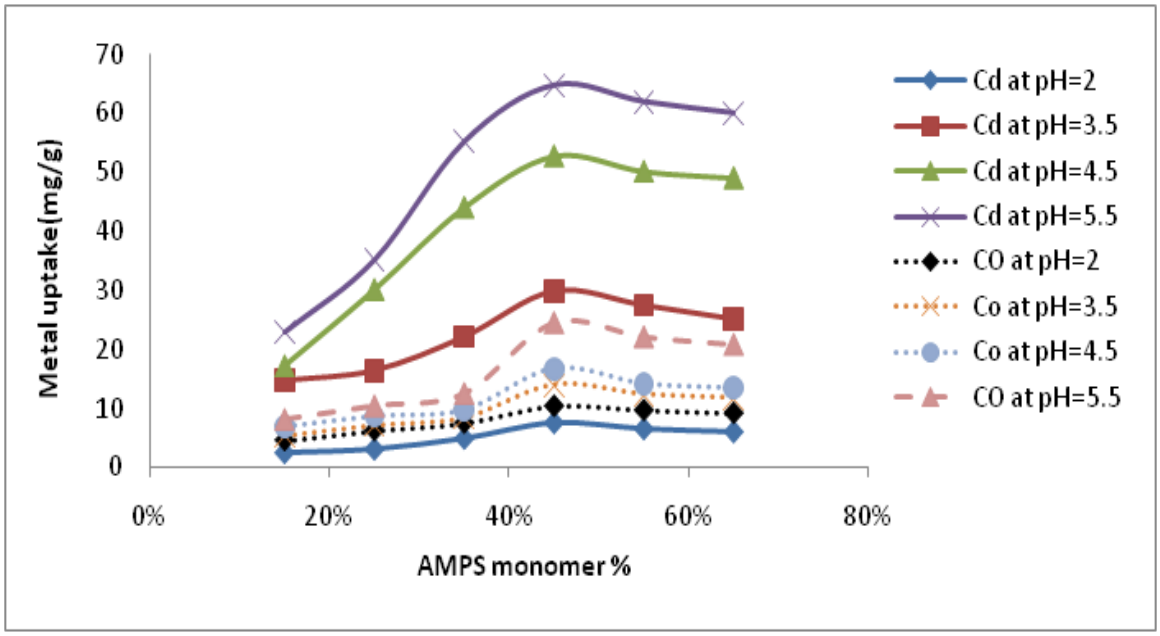


**Fig.14. Effect of AMPS % in grafting cotton–AMPS on Co (II) and Cd (II) uptake at various pH values.**

*Effect of Initial metal ion concentration (mg/l) on the removal of metal ions*

The effect of Initial metal ion concentration (mg/l) on the removal of Co(II) as for example by Un grafted cotton, Grafted cotton(AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ Nano magnetite) at 45% AMPS (conditions: pH=5.5, T=25°C; W/V = 0.5 /50 ml, t = 90 min was examined in Figure 15. It could be seen that the metal ion adsorption capacity increased with increasing metal ion concentration. This was due to a greater availability of the metal ions in the vicinity of the absorbent before the absorption–desorption equilibrium was reached. In addition, a higher initial concentration provides an important driving force to overcome all mass transfer resistances of the pollutant between the aqueous and solid phases, and thus increases the metal ion uptake [44]. Furthermore, it could be seen that the unmodified cotton can bind amounts of the metal ions due to the presence of functional groups such as hydroxyl, carboxyl, etc., which provide binding sites for the metal ions [45]. Moreover metal ions removal efficiency of Grafted cotton (AMPS+Cotton+ nano magnetite) is more than cotton (AMPS+Cotton) at the same conditions. This may be explained by nano particles increase the active site of cotton substrate by increasing its surface area and consequently increase the metal adsorptions.

*Effect of sorbent dosage on the removal of Cd (II) and Co (II) ions*

Effect of sorbent dosage on the removal of Cd(II) and Co(II) by Un grafted cotton, Grafted cotton(AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ Nano magnetite) at 45% AMPS (conditions: $C_0$ = 1200 mg/l ,pH=5.5, T=25 °C, t = 90 min) was studied at sorbent dosages from 0.5 to 2 g/50ml and represented in Figure 16. It was clear that Cd(II) and Co(II) ions uptakes increased by increasing sorbent dose reaching maximum at 2g/50ml. this might be attributed to high sorbent doses increased grafting of both AMPS monomer and nano magnetite particles on waste cotton substrate which hence increased the adsorption of metal ion uptakes in both metals.

*Effect of temperature of the medium on ions uptake*

Figure 17 showed the effects of temperature on the removal of metal ions by un grafted and grafted cotton sample with 8% magnetite nanoparticles & 45% AMPS content were studied at aqueous solution of pH 5.5 and at temperature range 20–50°C, and an initial metal ion concentration of 1200 mg/L. The elimination effectiveness for all tested samples increased with the increase in temperature from 20 to 50°C, revealing that the adsorption process was endothermic. On the other hand the removal efficiency for Cd ion (97%) at 50°C is much more than that of ions at the same condition (78%). One possible explanation was that the Co ion was well hydrated compared to cadmium ion. It had to lose part of hydration sheath in order to be adsorbed. This dehydration process of Co ions needed energy [46].

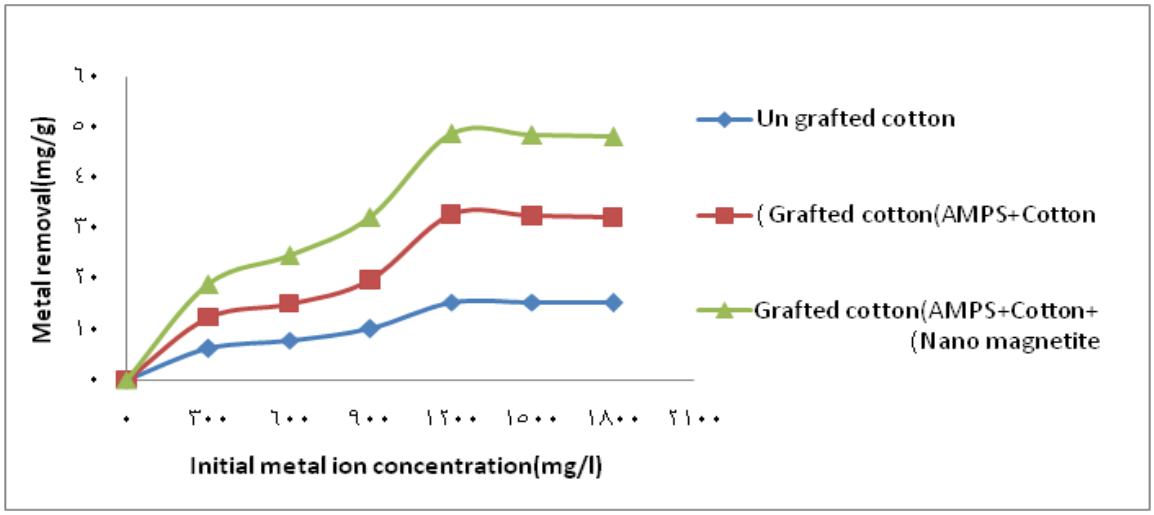


**Fig.15. Effect of Initial metal ion concentration (mg/l) on the removal of Co(II) by Un grafted cotton, Grafted cotton(AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ nano magnetite) at 45% AMPS (conditions: pH=5.5, T = 25°C; W/V = 0.5 /50 ml,t = 90 min).**

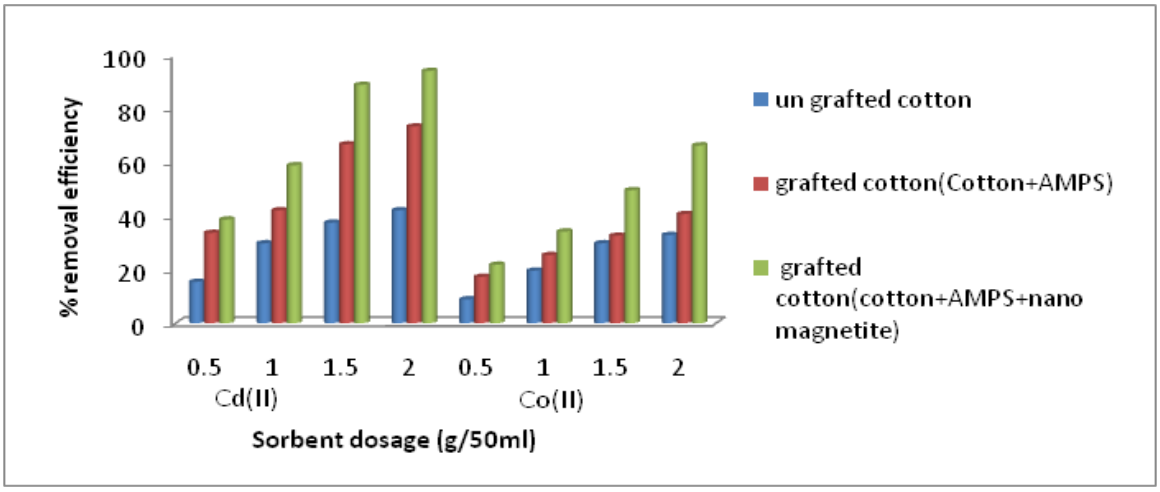


**Fig. 16. Effect of sorbent dosage on the removal of Cd(II) and Co(II) by Un grafted cotton, Grafted cotton(AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ nano magnetite) at 45% AMPS (conditions: $C_0$ = 1200 mg/l,pH=5.5, T=25°C, t = 90 min).**

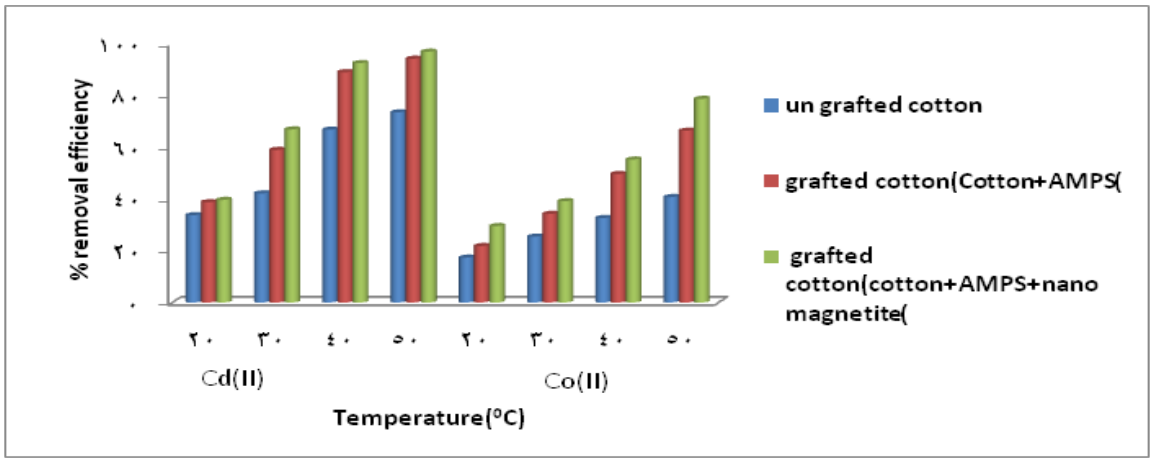


**Fig. 17. Effect of temperature on the removal of Cd(II) and Co(II) by Un grafted cotton, Grafted cotton(AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ nano magnetite) at 45% AMPS (conditions: $C_0$ = 1200 mg/l,pH=5.5, W/V = 0.5 g/50 ml, t = 90 min).**

*Treatment of waste water from cationic dyes*

The affinity of the modified grafted cotton fabrics to adsorb higher water content and its higher surface area with network structures allow dyes diffusing through its chains. As the modified cotton possesses ionic functional groups such as hydroxyl, carboxyl and amide groups, it could adsorb and trap cationic dyes and anionic dyes from wastewater [47].

Methylene blue (MB) is a cationic dye having various applications in chemistry, biology, medical science and dyeing industries. Its long term exposure could cause vomiting, nausea, anemia and hypertension [48]. Various physical, chemical and biological methods, including adsorption, bio sorption, coagulation/flocculation, advanced oxidation, ozonation, membrane filtration and liquid–liquid extraction had been widely used for the treatment of dye-bearing wastewater. The advantages and disadvantages of every removal technique have been extensively reviewed [49-51]. The removal of dyes in an economic way remains an important issue for researchers and environmentalists. Adsorption is a very effective separation technique in terms of initial cost, simplicity of design, ease of operation and insensitive to toxic substances.

Figure 18 represents the effect of initial dye concentration on the removal of MB by un grafted cotton, Grafted cotton (45%AMPS+Cotton) and Grafted cotton (45%AMPS+Cotton+ 8% Nano magnetite) (conditions: pH 8, T=25 $^{o}$C; W/V = 0.5 g /50 ml, t = 90min. It was observed that the adsorption capacity of the three tested samples increase with increasing dyes content. The adsorption capacity of Grafted cotton(45%AMPS+Cotton+8% nano magnetite) towards methylene blue dye higher than that of Grafted cotton (45%AMPS+Cotton) higher than that of un grafted cotton which may be due to the difference in their chemical structures. This was due to presence of magnetic nano particles besides the presence of the acid groups, amide groups –CONH and hydroxyl groups –OH in the AMPS and cotton chain which affect and promotes the adsorption capacity. The free carboxylic acid groups present have tendency to form salts with methylene blue cationic dye (basic dye), showing that the high affinity between the cationic dye and acid moiety is of chemical nature [52-54]. Also electrostatic interactions between the dye molecules and amide group via hydrogen bonding may be occurred [55].

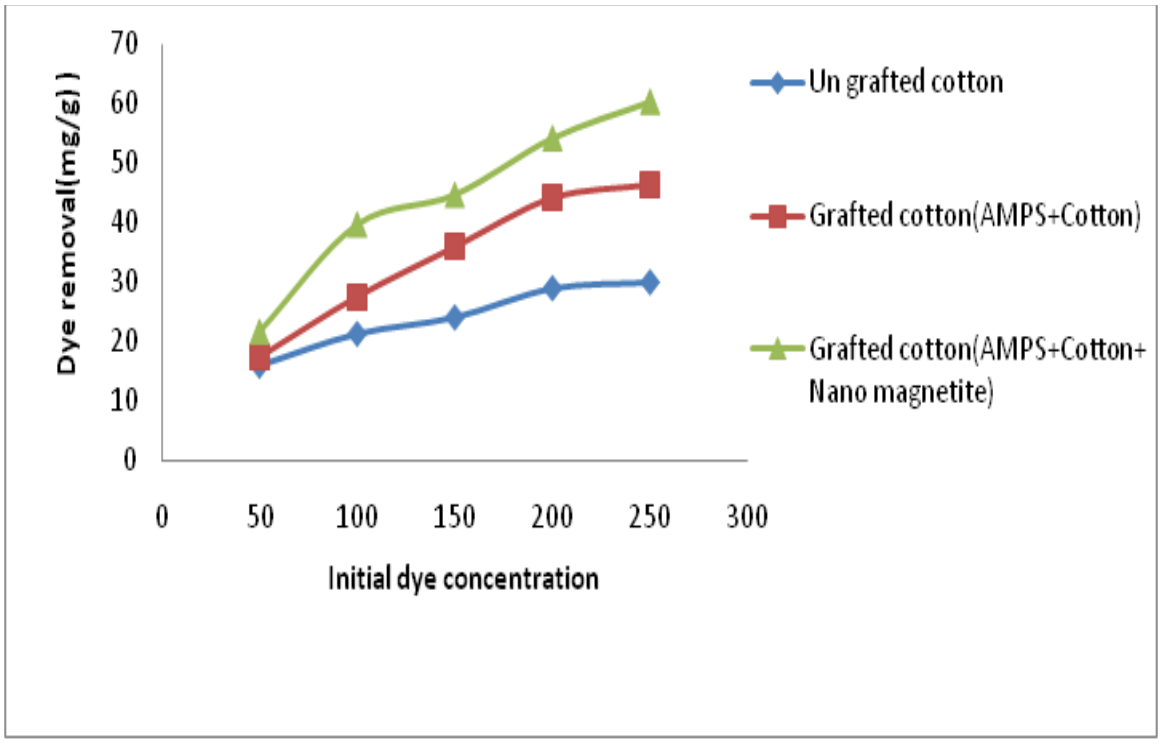


**Fig.18. Effect of initial dye concentration on the removal of MB by un grafted cotton, Grafted cotton Cotton) and Grafted cotton(AMPS+Cotton+ Nano magnetite) (conditions: pH 8, T=25 oC; W/V = 0.5 g).**

Figure 19 represents the effect of the AMPS concentrations on the adsorption capacity of selected sample of grafted cotton(AMPS+Cotton+8% nano magnetite) at various pH varying from 2-12 toward methylene blue (cationic) dyes. it was obvious that the adsorption capacity increased by increasing AMPS content reaching maximum at 45% and pH12. This may be attributed to the electrostatic interactions and salt formation between the dye molecules and AMPS moiety. Moreover Figure 20 represents the effect of pH on the removal of MB by un grafted cotton, grafted cotton(AMPS+Cotton) and grafted cotton (AMPS + Cotton+ nano magnetite) (conditions: $C_0$ = 100 mg/l, T=25°C; W/V = 0.5 /50 ml, t = 90min). From this figure one could conclude that, dye removal increase by increasing pH reaching maximum at pH12 because in alkaline media the affinity of salt formation with acid moiety increased the adsorption capacity. Consequently, Fig. 21 showed the effect of sorbent dosages on the removal of MB by un grafted cotton, grafted cotton(AMPS+Cotton) and grafted cotton(AMPS+Cotton+ nano magnetite) (conditions: $C_0$ = 100mg/l pH 12, T=25°C; t = 90min). It was observed that the adsorption of dye increased by increasing sorbent dose reaching equilibrium at 1.5 g/50ml according to the increasing of active site for methylene blue removal. Finally, Fig. 22 showed the effect of temperature on the removal of MB by un grafted cotton, Grafted cotton (AMPS+Cotton) and Grafted cotton (AMPS+Cotton+ nano magnetite) (conditions: $C_0$ = 100mg/l pH 12, W/V = 0.5 g/50 ml, t = 90min). It was clear that, methylene blue removal increased by increasing temperature reaching saturation at 50°C revealing that the adsorption process was endothermic. This might be attributed to the temperature increased the active site for grafted cotton substrate and consequently increased the dye removal.

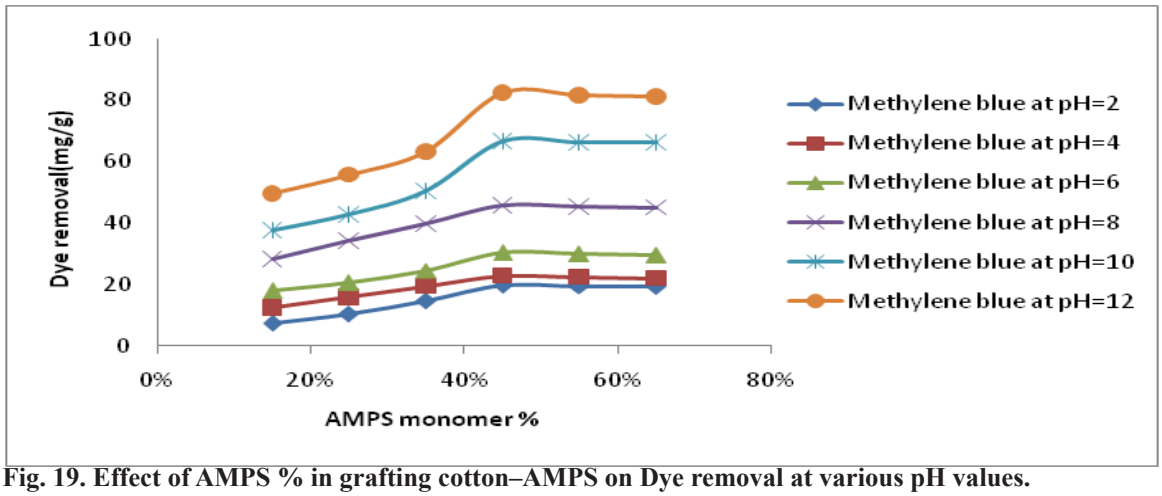


**Fig. 19. Effect of AMPS % in grafting cotton–AMPS on Dye removal at various pH values.**

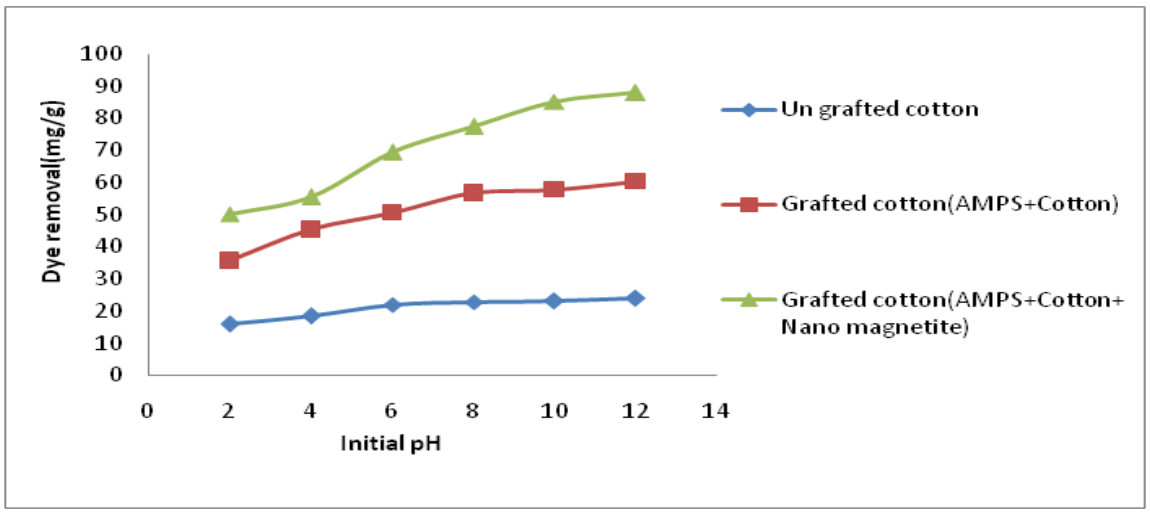


**Fig. 20. Effect of initial solution pH on the removal of MB by Un grafted cotton, Grafted cotton (AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ Nano magnetite) (conditions: C0 = 100 mg/l, T=25°C; W/V = 0.5 /50 ml, t = 90 min).**

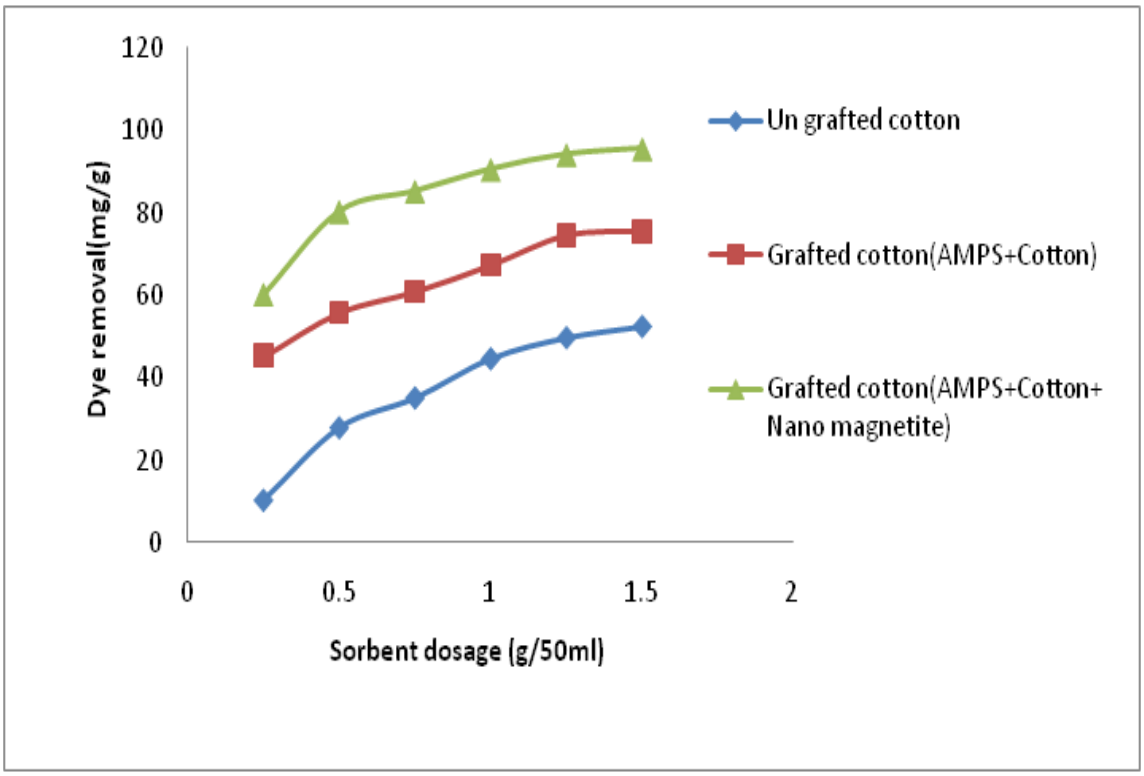


**Fig. 21. Effect of sorbent dosage on the removal of MB by Un grafted cotton, Grafted cotton(AMPS+Cotton) and Grafted cotton(AMPS+Cotton+ Nano magnetite) (conditions: $C_0$ = 100mg/l pH 8, T=25°C; t = 90 min)**

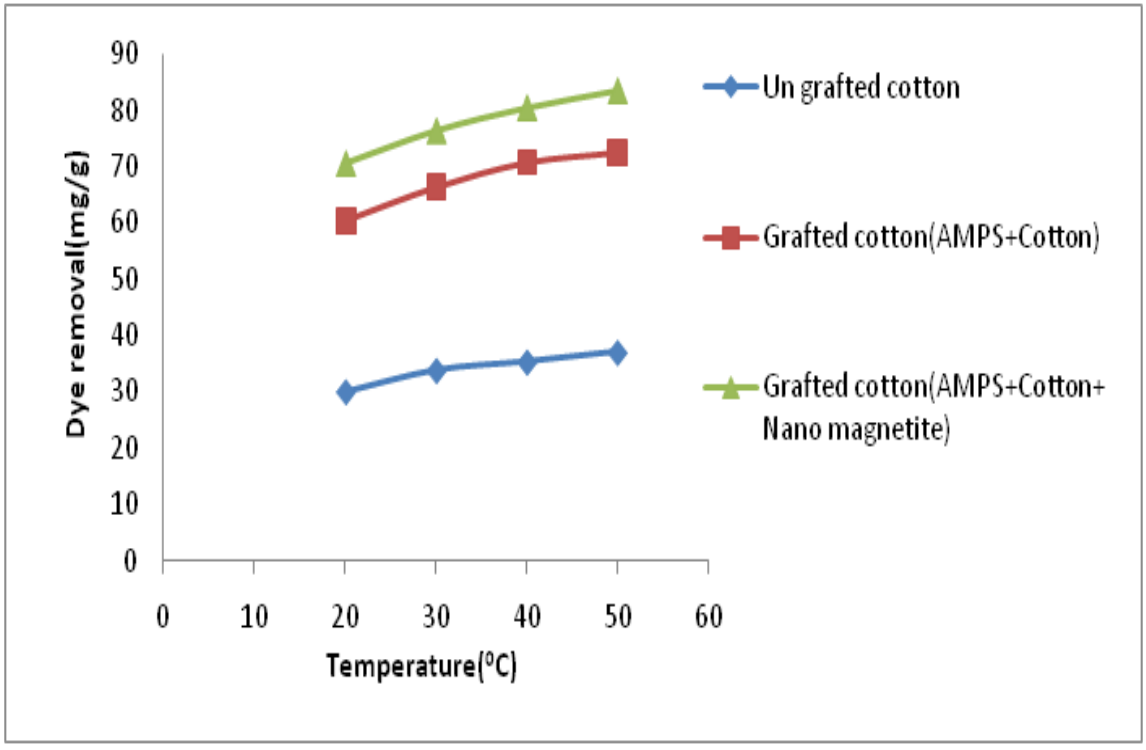


**Fig. 22. Effect of temperature on the removal of MB by Un grafted cotton, Grafted cotton (AMPS+Cotton) and Grafted cotton (AMPS+ Cotton + Nano magnetite) (conditions: $C_0$ = 100 mg/l, pH 8, W/V = 0.5 g/50 ml, t = 90 min).**

## Conclusions

The environmentally friendly modification of waste cotton surface fabric by (2-acrylamide-2-methyl propane sulfonic acid (AMPS) and nano magnetite particles were effectively synthesized using direct radiation grafting procedure. The results demonstrated that the prepared grafted nano magnetite cotton had higher grafting yield, grafting efficiency, swelling capacity and thermal stability than that of ungrafted cotton or grafted cotton with (AMPS). The green tested samples showed a great capability to remove metal ions such as $Co^{+2}$, $Cd^{+2}$ and methylene blue from their aqueous solution. The results discovered that the capability of the green modified cotton matrix is reliant on presence of nano magnetite particles and functional groups such as carboxyl; hydroxyl and amide moiety existing inside the grafted chains networks could capture these different kinds of pollutants. Moreover it depended on the size of the investigated metal ions and dyes as well as. The data revealed that the metal ions & dye adsorption capacity increased with increasing metal ion concentration, absorbed dose, PH of solution, reaching the maximum of metal uptake, 99%. Removal efficiency for all the green modified samples increased with increasing temperature from 30 to 50°C. Finally, increasing numbers of ionic groups in the chains of networks was known to increase their swelling capacity, metal ions adsorption & methylene blue removal capacity.

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# التعديل الأخضر لسطح نسيج القطن المهدر بواسطة (٢-أكريلاميد -٢-ميثيل البروبان حامض السلفونيك ، امبس) وجزيئات النانو المغناطيسية

**ريم كمال فرج و هند العايدي السعيد**

أ المعهد المصري لأبحاث البترول ، مدينة نصر ، القاهرة ، ١١٧٢٧ ، مصر

تم تحضير بوليمر أخضر( صديق للبيئة) يحتوي على مجموعات وظيفية محددة مع القدرة على امتصاص جرعات عالية من الأصباغ الملوثة والمعادن الثقيلة من المياه الملوثة. تم تحسين الخواص السطحية للأنسجة القطنية المهدورة من خلال تعديل السطح عن طريق التطعيم البلمرى باستخدام الاشعاع الجامى كوبلت ٦٠ لاكتساب خواص جديدة مثل الطابع المحب للماء أو غير المحب للماء على أساس التركيب الكيميائي للمونومر المستخدم. تم إجراء تعديل ألياف القطن السطحي للنيكلوفيلز عن طريق إدخال مونومر أنيوني (٢-أكريلاميد -٢ ميثيل حامض سلفونيك البروبان ، امبس) وجزيئات النانو المغناطيسية لتسهيل فصل المواد السامة بسهولة. العوامل التي تؤثر على التطعيم مثل تركيز مونومر ، تمت دراسة جرعة الإشعاع. تم توصيف واثبات خصائص سطح الهيكل للنسيج المطلي باستخدام حيود الأشعة السينية و مسح المجهر الإلكتروني و تحليل الثباتية الحراريةللوزن و الأشعة تحت الحمراء. أظهرت النتائج التي تم الحصول عليها كفاءة عاليةوامنةلازالةالكاديوم والكوبلت والأصباغ الكاتيونية (ميثيلين بلو) من المياه الملوثة.